RESEARCH AND TECHNOLOGIC WORK ON COAL AND RELATED INVESTIGATIONS, 1956

BY BUREAU OF MINES STAFF

* * * * * * * * * * Information Circular 7904

UNITED STATES DEPARTMENT OF THE INTERIOR
Fred A. Seaton, Secretary
BUREAU OF MINES
Marling J. Ankeny, Director

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RESEARCH AND TECHNOLOGIC WORK ON COAL AND RELATED INVESTIGATIONS, 1956[1]

by

Bureau of Mines Staff

INTRODUCTION AND SUMMARY

This report is the 21st in a series summarizing research and technologic work on coal and related investigations by the Federal Bureau of Mines - the third of the series published on a calendar-year basis.[2] Subject publications of the Bureau during 1956 are cited, and pertinent Bureau work is described briefly.

The most effective method of conserving our important coal reserves is through improved technology of production and utilization. Associated problems form the program of the Bureau.[3 4 5 6] Potential production is dependent on a knowledge of the quantity and quality of the reserves, and the Bureau continues to contribute information on this subject. Many problems are being studied that relate to improvements in current production, including mining methods and health and safety measures. Preparation of mined coal is necessary for its most efficient utilization. Preparation by conventional methods is being studied, as well as the development of new methods. Fundamental studies on the structure and properties of coal are necessary to insure full advantage of the special qualities of different coals. Controlling factors in the efficient use of coal in combustion, carbonization, gasification, and direct and indirect hydrogenation of coal are being studied continuously, and new processes are being investigated. The many advances in all these fields are discussed.

1/ Work on manuscript completed September 1958.

2/ Carman, E. P., Geer, M. R., and Riley, H. L., Report of Bureau of Mines Research and Technologic Work on Coal and Related Investigations, 1955: Bureau of Mines Inf. Circ. 7794, 1957, 103 pp.

3/ Bureau of Mines in cooperation with Bituminous Coal Research, Inc., Outlook and Research Possibilities for Bituminous Coal: Bureau of Mines Inf. Circ. 7754, 1956, 52 pp.

4/ Yancey, H. F., Coal: Min. Eng., February 1956, vol. 8, No. 2, pp. 195-196.

5/ Cohn, E. M., and Perry, Harry, Activities and Publications of the Bureau of Mines: Jour. Chem. Ed., vol. 33, No. 7, July 1956, pp. 331-333.

6/ Staff, Bureau of Mines, Mineral Facts and Problems: Bureau of Mines Bull. 556, 1956, pp. 37-49, 115-135, 445-456.

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This work was performed at the following Bureau stations:

Station	Location	Main coal activities
Headquarters.....	Washington, D. C.	Programing and budgeting, foreign activities, Government fuel-engineering service.
Anthracite Experiment Station....	Schuylkill Haven, Pa.	Anthracite investigations.
Region I:		
Alaska Mining Experiment Station	Juneau, Alaska	Exploration.
Anchorage Experiment Station....	Anchorage, Alaska	Sampling and analysis.
Northwest Experiment Station....	Seattle, Wash.	Preparation.
Region III:		
Denver Experiment Station.....	Denver, Colo.	Western coals investigations.
Lignite Experiment Station.....	Grand Forks, N. Dak.	Lignite investigations.
Region V:		
Central Experiment Station.....	Pittsburgh and Bruceton, Pa.	Health and safety investigations (District B); mining, preparation, combustion, carbonization, hydrogenation, petrography and constitution, sampling and analysis of bituminous coal.
Appalachian Experiment Station..	Morgantown, W. Va.	Gasification, low-temperature tar.
Eastern Experiment Station.....	College Park, Md.	Boiler-water research and service, stream and air pollution.
Southern Experiment Station.....	Tuscaloosa, Ala.	Preparation and carbonization.
Gorgas Experiment Station.....	Gorgas, Ala.	Underground gasification.

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Among the services furnished to Government agencies are sampling and analysis of coal from current production; the results are used by Federal agencies in awarding contracts. During the year, 649 tipple samples were collected at 253 mines in 12 States. Assistance also is given in establishing standardized sampling procedures for coal delivered to Government installations. The volume of analytical work is indicated by the fact that in 1956 over 326,000 chemical and physical tests were made on more than 20,000 samples.

Since World War II, international trade in coal has increased greatly, and this has emphasized the need for international classification of coal. The Bureau has cooperated with the Coal Committee of the Economic Commission for Europe in establishing the system adopted in 1956 for classification of coals higher in rank than lignite and is continuing collaboration in establishing an international classification of brown coals and lignite.

The quality of coal is determined by the results of analysis. Need for revising standard methods is recognized, and methods for determining ash and mineral matter are being studied. New methods are being developed for determining chlorine and volatile matter in coal and for measuring the amount of incombustible material in coal-mine dust: The first is especially significant when selecting coal to minimize formation of bonded deposits in furnaces; the second, for compliance with safety regulations.

Methods for studying the structure and chemistry of coal have been extended and improved in recent years. Optical properties, including indexes of refraction and absorption, and infrared, visible, ultraviolet, and X-ray spectra have proved to be useful tools. The fine physical structure of coals has been studied by means of adsorption to determine pore diameters. Details of differences in the physical and chemical properties of the petrographic constituents of coal are being studied. Two new methods of mild chemical treatment of coal have been developed and offer considerable promise of advancing knowledge of the chemical structure of coal. Another mild treatment, microbial degradation of coal, also has yielded some structural information.

The Bureau has participated in a program to develop Alaskan coal reserves. A major activity has been drilling to delineate the reserves in the Wishbone Hill District of the Matanuska field - the major source of coal for the Anchorage area.

The use of the pneumatic coal planer and the scraper-shaker loader is being studied in the anthracite fields; the results are encouraging. Cleaning-plant refuse, packed pneumatically, offers a means of roof support in mining anthracite. These are but two of the current programs to improve the efficiency of mining operations and to reduce mining costs. The Bureau also is actively cooperating in developing adequate mine-drainage control.

Bituminous coal-mining studies included methods and equipment for underground development, longwall mining, systems and equipment for face haulage, and augers for surface mining. Costs of timber and roof-bolt supports were compared. Records are being examined to determine the recovery of coal from different operations - an important item when estimating useful reserves.

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During 1956, reports were published on coking-coal reserves in 13 counties in Pennsylvania, Tennessee, and West Virginia, where fieldwork has been completed.

Bureau representatives were requested to aid in coal projects in Afghanistan, Colombia, and India in 1956; funds were made available by the International Cooperation Administration (ICA).

The importance of the health and safety activities of the Bureau cannot be overemphasized. Both human and mineral resources thus are conserved. Collecting and publishing statistics of accidents and fatalities pinpoint areas where further research in mining methods is needed. Attention may be called to recent studies of roof-bolting and electrical equipment in mines. The program of safety training and education is a continuing one.

Mined coal may have to be stored before it is used, and the period of storage varies greatly. Storage of lignite is of greater concern than storage of bituminous coal. Observation by the Bureau since 1953 of eight piles of lignite (over 2 million tons), recovered and stored during construction of the Garrison Dam in North Dakota, have yielded significant information on storage of lignite. The annual decrease in heating value has been less than 1 percent; the major problem has been erosion by wind and rain.

Because of severe winter weather and the inherently high moisture content of lignite, freezeproofing is an important problem. A reproducible method for determining the effectiveness of freezeproofing agents has been developed.

Preparation of coal has become increasingly important since mechanical mining methods were adopted. Cleaning and dewatering fine coal are the most difficult aspects of coal preparation, and these subjects are receiving intensive attention. Cleaning coarse coal by the use of dense mediums also is being studied. The work of the Bureau includes a survey by counties of the preparation of coking coals and the reduction of ash in anthracite. A special study showed that low-ash coke can be prepared by carbonizing extracts of certain bituminous coals; hence bituminous coal may become an alternative source of electrode carbon.

Despite the fact that combustion has been the major use of coal for centuries, scientific and engineering problems remain to be solved for its most efficient use. One of these problems is heat transfer in furnaces and its dependence on flame temperature and ash deposits on the external surfaces of boiler tubes. Data from commercial installations are being analyzed, and the nature of the deposits studied. The fluid mechanics involved in furnace operation are not understood fully yet, and this subject also is being studied.

The Bureau provides fuel-engineering services to other Federal agencies to increase their efficiency in the use of coal. One of these services involves recommendations on boiler-water treatment to minimize difficulties arising from condensate corrosion.

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Combustion releases materials that contribute to air pollution. Methods of reducing this nuisance are being studied by the Bureau. Incinerators for disposing of normal and radioactive wastes have been developed. The removal of sulfur dioxide from fuel gases is a continuing project.

Bureau studies of carbonization of coal include the mechanism of thermal decomposition, process variables, and assay tests by both low- and high-temperature methods. A fluid-bed unit has been built to produce primary tar, which then may be passed through a cracking unit maintained at controlled higher temperatures to study conditions that determine the composition of tar obtained in conventional coke ovens. Dependence of coke structure on the petrographic composition of the coal used is being studied. The effects of inerts in coal, of pretreatment of coal below its softening temperature, and of the properties of the resulting coke are being studied.

A carbonization assay (BM-AGA), developed many years ago, is being used for assessing the value of coking-coal reserves. Six additional reports on coals from counties in the Appalachian region were published in 1956. Results of the assay closely correlated commercial experience in a cooperative study with the Canadian Department of Mines and Technical Services. Coking of western coals in a slot-type oven was studied in cooperation with steel companies. An oven of similar design has been used to study the effects of flue temperature and bulk density on coking.

Anthracite is being used for metallurgical fuel, principally in blends with bituminous coal. Current studies may extend this use, especially as a cupola fuel. Calcining anthracite to increase its resistance to thermal shock is one of the main subjects being studied.

Carbonization of coal at temperatures lower than those used in conventional practice produces a char more reactive than blast-furnace coke and also produces increased yields of tar that may prove to contain useful chemicals. A survey of results obtained in a low-temperature assay of North-Central U. S. lignites was published in 1956. Studies of drying and carbonizing fine coal in an entrained state were continued and extended to a lignite from India. The effect of carbonizing uraniferous lignites at low-temperatures before extraction was studied. To anticipate difficulties that might be encountered when lignite is gasified by pressure, carbonization of lignite at elevated pressures is being studied.

Tar obtained under the relatively mild conditions of low-temperature carbonization reflects the composition of the original coal to a much greater extent than tar from conventional high-temperature coking. Separation and characterization of the constituents of low-temperature tars from lignite and bituminous coals are included in the Bureau program. Upgrading certain fractions to yield materials of commercial interest is being studied.

The importance of gasification of coal will increase as the reserves of natural gas and petroleum decrease. Hydrogen is required for direct hydrogenation of coal to liquid products, and mixtures of hydrogen and carbon monoxide are needed for indirect hydrogenation and high-B.t.u. gas. Studies

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are progressing on the mechanisms of gasification reactions and on problems of operating various types of gasifiers. Producing hydrogen by the steam-iron process - iron is alternately reduced by producer gas and oxidized by steam - is receiving attention. Several studies relate to the possibility of applying nuclear energy to supply the heat required for coal gasification; materials of construction are a major problem.

For direct use or synthetic purposes, gas from coal must be purified to remove gaseous and solid constituents detrimental to the desired objective. Methods of purification are being investigated.

Gasifying coal underground and bringing the energy to the surface, without the need for mining coal, has been an intriguing subject for many years. Studies of hydraulic fracturing and electrolinking in the bed are progressing.

The two principal methods for converting coal to fluid fuels are: Direct reaction of coal with hydrogen at elevated temperatures and pressures; and gasification of coal to mixtures of carbon monoxide and hydrogen, followed by catalytic transformation of these gases to the desired products. Both methods are being studied on scales ranging from fundamental laboratory work to pilot-plant operation. In direct coal hydrogenation the autoclave is usually the "test tube" used first to evaluate catalysts and process variables. Such studies are being continued. Developing methods of characterizing the products is an important part of this work. Pilot-plant work has been concerned with the conditions for maximum production of distillate fuels at 1,500 to 3,500 p.s.i.g. and hydrogenating coal at the very high temperature of 800° C. for short residence times.

In the synthesis of liquid fuels from carbon monoxide and hydrogen (Fischer-Tropsch synthesis) major problems are encountered in preparing catalysts that have a satisfactory life under the conditions of operation, are resistant to poisons, and yield products of a desired composition. These problems are being studied. Knowledge of the chemistry of metal carbonyls has contributed significantly to an understanding of the reaction. The Fischer-Tropsch synthesis releases large amounts of heat; this fact introduces major problems in designing reactors so that temperature may be controlled and the evolved heat used effectively. Recent work, on a pilot-plant scale, indicated that a gas-recycle process offers considerable promise.

In cooperation with the University of West Virginia (Morgantown) the Bureau is conducting a program of mutual assistance to educate and train scientific and engineering students. During this 10th year of the student-trainee program, approximately 50 undergraduates were employed on part-time jobs. In addition, eight graduates worked toward advanced degrees by completing Bureau research projects at the Appalachian Experiment Station. This program provides financial assistance and on-the-job training to science students and at the same time considerably alleviates the manpower shortage in the Bureau. It also serves to induce superior graduates to continue in Government employment. Over the years more than 250 graduate and undergraduate students have participated in this program, and many have remained in Government service.

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Boslego Coal Co.
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Carbone Corp.
Carborundum Co.
Colorado Fuel and Iron Corp.
Columbia-Geneva Steel Div., U. S. Steel Corp.
Comision de Fomento Industrial, Cali, Colombia
Eastern Gas & Fuel Associates
Electro Metallurgical Co., Division of Union Carbide Corp.
El Paso Natural Gas Co.
Esso Research & Engineering Co.
Fansteel Metallurgical Corp.
Ford Motor Co.
General American Transportation Corp.
Glen Alden Coal Co.
Grafton Coal Co.
Great Northern Railroad Co.
Harvard School of Public Health
Heyl and Patterson, Inc.
Hudson Coal Co.
Island Creek Coal Co.
Johns-Manville Co.
Kaiser Steel Corp.
M. W. Kellogg, Co.
Kemmerer Coal Co.
Madras State Government (India)
Mine Safety Appliances Co.
Ministry of Production, Central Government of India
Minnkota Power Cooperative, Inc.
National Carbon Co., Division of Union Carbide Corp.
National Lead Co. of Massachusetts
Northern States Power Co.
Northwestern Improvement Co.
Norton Co.
Petrocarb Equipment, Inc.
Pennsylvania State Department of Forests and Waters
Pennsylvania State Department of Mines and Mineral Industries
Pine Township Coal Co., Inc.
Pittsburgh Consolidation Coal Co.
Quality Excelsior Coal Co.
Reading Anthracite Co.
Research Foundation, Colorado School of Mines
Riley Stoker Corp.
Royal Afghan Ministry of Mines, Kabul, Afghanistan
Sahara Coal Co.
Southern Coal & Coke Co.
Special Air-Pollution Study of Louisville & Jefferson County (Ky.)
Susquehanna Collieries Co.
U.S. Department of State (Neyveli, India, project)
Texas Power & Light Co.
Union Carbide Corp.
Union Pacific Coal Co.
University of Alabama
University of North Dakota
University of Washington
U.S. Army, Corps of Engineers
U.S. Atomic Energy Commission
U.S. Geological Survey
United States Pipe & Foundry Co.
U.S. Public Health Service
United States Steel Corp.
Western Precipitation Corp.
West Virginia University
Wyodak Coal Resources Development Corp.

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ORIGIN AND PROPERTIES OF COAL AND RELATED MATERIALS

Inspection, Sampling, and Analysis

The Bureau furnishes the following services to Government agencies and the general public:

1. Reporting authoritative analyses of coal to Federal agencies, advising them on technical matters relating to coal purchases, and assisting them in determining awards of contracts and in carrying out the terms of the contracts.

2. Conducting sampling surveys at mines to obtain representative analyses of coal and other information for use in connection with Government coal purchases, surveys of coal reserves, and for other special purposes.

3. Checking coal-sampling methods at Government installations and instructing Federal personnel in proper sampling methods.

4. Making available to Federal agencies and the general public representative analyses and other data on American coals by publications and by maintaining a file of coal analyses.

These services have resulted in substantial savings to the Government by making possible coal-purchase contracts on a guaranteed-quality basis, adjustment of prices when shipments fall below contract specifications, and a basis for evaluating coal bids. Certain facilities are also extended to State agencies and, through ICA, to foreign countries.

Requests from Federal agencies for analyses for use in evaluating coal bids continued at about the same rate as in the previous year. Recommendations were made for awards of contracts by some agencies.

During the year 649 tipple samples were collected at 253 mines in 12 States; 38 carbonization samples were collected at mines in Kentucky and West Virginia. Under a working agreement with the Atomic Energy Commission (AEC), 608 samples of delivered coal were collected at the Savannah River Operation plants, Aiken, S. C., and 575 samples at the Dana plant, Ind.

At the request of the Marine Corps, Camp Lejeune and Cherry Point, N. C., were visited January 12 and 13, to observe coal-sampling procedures and to instruct personnel in proper inspection and sampling procedures. Meetings were held with representatives of the various Army commands at Washington, D. C., February 13-17, to review coal-sampling techniques and coal requirements for the fiscal year 1957. At the request of AEC, the Oak Ridge, Tenn., plant was visited to discuss their coal-sampling and procurement program.

Alaska was visited October 1 to 20 to review coal-sampling procedures at the Army and Air Force installations. All operating coal mines in the fields near the Alaska Railroad were examined for the Naval Fuel Supply Office, which

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handles procurement for Army and Air Force bases in Alaska. From these data and consultations with the operators, estimates were prepared of developed reserves and production potentials of the region. Special examinations and reports on individual operations also were made for the Naval Fuel Supply Office.

Three of a series of annual publications were released on analyses of tipple and delivered samples of coal collected throughout the United States and Alaska.[7] [8] [9] The analyses, arranged alphabetically by States, counties, towns, and mines, comprise proximate analysis, ultimate analysis, calorific value, ash-softening temperature, agglomerating index, free-swelling index, and Hardgrove grindability index.

From January 1 through December 31 more than 36,000 samples of coal, coke, char, tar, coal-mine dust, and related materials were analyzed. Included in this number were 20,438 samples collected during 3,723 inspections in 20 States and Alaska, in accordance with provisions of Public Law 552, the Federal Coal-Mine Safety Act; 11,592 representing Government coal purchases and tipple inspections (54 percent from branches of the Department of Defense; and 4,024 from the Federal Bureau of Mines and other government organizations conducting research and exploration projects on coal. Over 326,000 chemical and physical tests were made on these samples.

Total silica was determined for 50 samples of rock dust collected by Federal coal-mine inspectors to determine compliance with Public Law 552, which specifies that rock dust used in coal mines shall contain not more than 5 percent total silica. Samples of 17 other rock dusts were examined for total silica content, size, and caking properties, in connection with investigations of methods for wet rock dusting.

A power failure caused by an extraordinarily heavy snowstorm early in January resulted in considerable damage to the Alaskan coal laboratory. During repairs and subsequent personnel changes, the analytical work was interrupted; but samples were crushed, quartered, sealed, and shipped by airmail to the Pittsburgh laboratory to minimize delays. A total of 480 samples was analyzed, including 455 samples of coal from the 6 mines that supply the Army and Air Force bases.

7/ Aresco, S. J., Haller, C. P., and Abernethy, R. F., Analyses of Tipple and Delivered Samples of Coal (Collected During the Fiscal Year 1953): Bureau of Mines Rept. of Investigations 5085, 1955, 82 pp.

8/ Aresco, S. J., Haller, C. P., and Abernethy, R. F., Analyses of Tipple and Delivered Samples of Coal (Collected During the Fiscal Year 1954): Bureau of Mines Rept. of Investigations 5221, 1956, 77 pp.

9/ Aresco, S. J., Haller, C. P., and Abernethy, R. F., Analyses of Tipple and Delivered Samples of Coal (Collected During the Fiscal Year 1955): Bureau of Mines Rept. of Investigations 5270, 1956, 66 pp.

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International Classification of Coal

Increased world trade in coal since World War II necessitated the development of international methods of analyzing,[10/] testing, and classifying coals to provide a common language for evaluating coals of various countries. In the past, coal-classification schemes used in different countries were developed in accordance with national needs and characteristics of domestic coals without reference to coals of other countries. Terms for describing similar or identical coals differed in each country, which caused confusion when evaluating coals for international trade and coal-research programs. To overcome these difficulties the Coal Committee of the Economic Commission for Europe in 1949 established a Classification Working Party to make recommendations concerning an international classification system. Experts appointed from nearly every European country and the United States have participated in the work. A representative of the Bureau of Mines has served as a delegate to nearly all meetings of the committee.

The Classification Working Party has made much progress in developing an international classification system. In 1953 systems for an international classification of hard coals by type and a statistical grouping of hard coals were formulated and then tried for 2 years; no major difficulty was encountered. The Coal Committee, at its 37th session in March 1956 noted that the trials had been successful and recommended practical application of the systems.

Figure 1 shows the international system for classification of hard coals by type. The term "hard" coal, according to European usage, refers to coals higher in rank than lignite and brown coal. The term "type" refers to differences in the degree of coalification in the natural series peat to anthracite. Superimposed on this classification is a system of statistical groups of hard coals, in which the subgroups are combined into a relatively small number of statistical groups.

A classification system for brown coals and lignites is being developed. At the 13th session of the Classification Working Party in June 1956 the lower limit of class 9 hard coals was fixed at 5,700 kcal./kg. (10,260 B.t.u. per pound) on the moisture- and ash-free (m.a.f.) basis, which in effect becomes the dividing line between hard coals on one hand and brown coals and lignites on the other. To establish parameters for classification of brown coals and lignites, an exchange of samples was arranged among several cooperating laboratories. In the exchange the Bureau of Mines analyzed 29 samples of low-rank coals from Czechoslovakia, the Federal Republic of Germany, Poland, Sarawak, Union of Soviet Socialist Republics, the United Kingdom, the United States, and Yugoslavia. They supplemented similar analyses made previously on low-rank coals from other sources. Determinations included moisture in as-received and air-dried coal by toluene distillation, tar by Fischer-Schrader, low-temperature carbonization assay, moisture-holding capacity by equilibration at 97 percent relative humidity and 30° C., rational analysis including bitumens and humic acids, and proximate and ultimate analyses.

10/ Selvig, W. A., Fourth International Meeting on Coal and Coke: Magazine of Standards, vol. 26, No. 9, 1955, pp. 282-283.

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GROUPS (determined by caking properties)			CODE NUMBERS										SUBGROUPS (determined by coking properties)		
GROUP NUMBER	ALTERNATIVE GROUP PARAMETERS		The first figure of the code number indicates the class of the coal, determined by volatile-matter content up to 33% V. M. and by calorific parameter above 33% V. M. The second figure indicates the group of coal, determined by caking properties. The third figure indicates the subgroup, determined by coking properties.										SUBGROUP NUMBER	ALTERNATIVE SUBGROUP PARAMETERS	
	Free-swelling index (crucible-swelling number)	Roga index												Dilatometer	Gray-King
3	> 4	> 45				334	435	535	635				5	> 140	> G8
							∇A	∇B	∇C				4	> 50-140	G5-G8
						333	434	534	634				3	> 0-50	G1-G4
							433	533	633	733					
						332 a	332 b	432	532	632	732	832	2	⩽ 0	E-G
										∇D					
2	2½-4	> 20-45				323	423	523	623	723	823		3	> 0-50	G1-G4
						322	422	522	622	∇IA 722	822		2	⩽ 0	E-G
						321	421	521	621	721	821		1	Contraction only	B-D
						IV				∇IB					
1	1-2	> 5-20			212	312	412	512	612	712	812		2	⩽ 0	E-G
					211	311	411	511	611	711	811		1	Contraction only	B-D
						III				∇II					
0	0-½	0-5		100 I A B	200 II	300	400	500	600	700	800	900	0	Nonsoftening	A
CLASS NUMBER →			0	1	2	3	4	5	6	7	8	9	As an indication, the following classes have an approximate volatile-matter content of: Class 6 33-41% volatile matter; 7 33-44% " "; 8 35-50% " "; 9 42-50% " "		
CLASS PARAMETERS	Volatile matter (dry, ash-free) →		0-3	> 3-10 (>3-6.5 / >6.5-10)	> 10-14	> 14-20	> 20-28	> 28-33	> 33	> 33	> 33	> 33			
	Calorific parameter [a] →		—	—	—	—	—	—	> 13,950	>12,960-13,950	>10,980-12,960	>10,260-10,980			
CLASSES (Determined by volatile matter up to 33% V. M. and by calorific parameter above 33% V. M.)															

Note: (i) Where the ash content of coal is too high to allow classification according to the present systems, it must be reduced by laboratory float-and-sink method (or any other appropriate means). The specific gravity selected for flotation should allow a maximum yield of coal with 5 to 10 percent of ash.
(ii) 332a . . . >14-16% V. M.
332b . . . >16-20% V. M.

[a] Gross calorific value on moist, ash-free basis (30° C., 96% relative humidity) B. t. u./ lb.

FIGURE 1. - International Classification of "Hard" Coals.

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Analytical Procedures

Determination of Mineral Matter

A method developed in Germany for direct determination of mineral matter in coal was investigated for application to American coals. Empirical formulas generally have been used for correcting ash to mineral matter when calculating coal analyses to a pure-coal basis, that is, coal free from its ash-forming mineral matter. In formulas of this type, where the calculation of mineral matter is based upon an average factor to correct for water of constitution of the shaly material, the selected factor is arbitrary and may be in error for a particular coal, but is of minor importance when the amount of residual ash after acid treatment is small.

The direct method is based upon eliminating most of the mineral matter by treating the coal with hydrofluoric and hydrochloric acids. Mineral matter is calculated from loss in weight, and corrections are made for residual ash, pyrite not removed by the acid treatment, and chlorine taken up by the coal.

Pretreatment with hydrochloric acid of coals containing mineral carbonate reduced the residual ash considerably, because formation of insoluble calcium fluoride was minimized. Furthermore, a correction for sulfur retained as sulfate in the ash of the acid-treated coal gave improved results. Methods were studied for reducing the chlorine content of the treated coal.

Coal substantially free of mineral matter would also be desirable for studying many other properties and reactions of coal, that is, density, X-ray and reflectance measurements with little or no correction due to mineral matter; carbonization, gasification, and hydrogenation of coal in the absence of possible catalytic effects of the mineral matter; and direct determination of oxygen.

Determination of Ash

Chemical analyses were published of 323 samples of ash from bituminous, subbituminous, and lignitic coals from 24 States and Alaska.11/ Knowing the composition of the coal ash is useful when studying the clinkering and slagging characteristics of coals burned in fuel beds. It also aids in predicting the flow properties of slag in slag-tap furnaces fired by pulverized coal or in the more recently developed cyclone furnaces using crushed coal. The behavior of slag in such furnaces can be evaluated more reliably from ash-composition slag-viscosity relationships than from cone-fusion temperatures. For the same reason, the information is of interest for designing and operating recently developed equipment for complete gasification of pulverized coal with oxygen and steam to produce synthesis gas. Ash composition also is important when selecting coals for special purposes, such as for manufacturing and burning the finer grades of ceramic ware.

11/ Selvig, W. A., and Gibson, F. H., Analyses of Ash From United States Coals: Bureau of Mines Bull. 567, 1956, 33 pp.

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In addition to the ash analyses the compilation includes a discussion of the nature and occurrence of ash-forming mineral matter in coal and the relation of ash composition to fusibility of ash.

Coal ashes from bituminous coals of the United States vary widely in chemical composition but generally are within the following limits: Silica (SiO_2), 20 to 60; alumina (Al_2O_3), 10 to 35; ferric oxide (Fe_2O_3), 5 to 35; calcium oxide (CaO), 1 to 20; magnesia (MgO), 0.3 to 4; titania (TiO_2), 0.5 to 2.5; alkalies ($Na_2O + K_2O$), 1 to 4; and sulfur trioxide (SO_3), 0.1 to 12 percent.

Comparable limits for coals of subbituminous and lignitic rank cannot be given because of the relatively small number of analyses available; however, subbituminous coals may contain more CaO, MgO, and SO_3 than bituminous coals. This trend is even more pronounced for lignite ash, and SiO_2 and Al_2O_3 generally are lower than in ash from bituminous coals.

New Analysis Procedures

Chlorine in Coal

In England certain correlations have been made between chlorine in coal and corrosion of boiler tubes. A project therefore was started to determine chlorine in those coals of the United States that are normally received for analysis. Several possible methods were compared. The one adopted for the survey consists of burning coal with oxygen under pressure in a calorimeter-type bomb and determining chlorine in the washings by electric titration with standard silver nitrate. At the end of the period covered by this report, approximately 75 samples from several States had been analyzed.

Volatile Matter in Coal

The details of standard procedure for determining volatile matter are rather vague; the results for certain lower rank coals, chars, and an occasional unusual anthracite vary considerably. From the various procedures submitted to the International Standardization Organization, the French-Belgian, or double-crucible, method appeared applicable to all ranks of coal. Because previous tests showed that the rate of heating was the most critical of the several variables in this empirical test, this rate was determined and applied to the regular standard procedure and equipment. As all tests on low-rank coals were satisfactory, this method has been adopted for routine testing. It reduces the time of testing by 20 percent, and reproducibility is far superior to that of the standard procedure.

Coal-Mine Dusts

The incombustible content of coal-mine dusts has been determined for many years by the same time-consuming method of analysis as used for coal. A modification of the rapid volumeter method, based upon the specific gravity of a coal dust-limestone mixture, has now been developed. All samples from Title II mines tested by this method and showing between 50 and 75 percent

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incombustible material are checked by the chemical method. Samples from Title I mines are tested exclusively by this rapid method. Since February 1956, 18,760 samples have been tested by the volumeter method, and only 32.4 percent of these had to be analyzed chemically. This screening procedure resulted in a considerable saving, which allowed testing of more samples. If all the samples received during the year had been analyzed chemically, an additional $20,000 or more would have been required.

Structure and Chemistry of Coal and Related Materials

The Bureau program of fundamental research on coal and carbonaceous materials and their products was expanded. A more fundamental examination of the nature of the petrographic components of coal was begun by separating the finely divided maceral constituents to provide more homogeneous material for basic studies, including helium and mercury densities, gas adsorption, diffraction, and optical techniques. Thermal studies will be made of coal components, graphite, and other carbonaceous materials.

Indexes of Refraction and Absorption

Indexes of refraction and absorption are useful in studies of the molecular structure of coals. These optical constants have been determined for vitrinite (the major and most homogeneous component) of different ranks of coal by determining its microscopic reflectivity in several media. Reports of similar work have appeared recently in the literature with divergent results that suggested lack of objectivity in some procedures; hence the present work was done with sensitive photoelectric equipment.

The maximum index of refraction increased rather slowly with rank in the lower rank coals, then more rapidly until it reached an apparent maximum in anthracite approaching that of graphite. The minimum index varied similarly, except that it appeared to decrease in the highest rank anthracites. The index of absorption increased slowly in the lower ranks then very rapidly in the anthracites. That the anisotropy parallel and perpendicular to the bedding plane was low in the younger coals and increased markedly with rank may be explained by the approach of the structure of anthracite to the layer plane structure of graphite.

Infrared, Visible, and Ultraviolet Spectra

That the electronic absorption exhibited by bituminous coals in the infrared region may not be attributable to polynuclear condensed aromatics has been discussed.12/ The lack of both strong absorption intensity and fine structure in the ultraviolet and visible regions of the spectrum places severe limitations on the amounts of polynuclear condensed aromatics that can be present. The color of coal is unattributable to such aromatics even if present. Paramagnetic resonance measurements in European laboratories have shown that free radicals are present in coal.

12/ Friedel, R. A., and Queiser, J. A., Infrared Analysis of Bituminous Coals and Other Carbonaceous Materials: Anal. Chem., vol. 28, No. 1, 1956, pp. 22-30.

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Correlations between color and free radical content have been determined for high-volatile bituminous coals. Free radicals might explain not only the color of coal but also its electronic absorption in the infrared, its comparatively weak and nonspecific absorption in the visible and ultraviolet spectra, its refractive and absorptive indices, and its reflectance.

X-ray Diffraction

X-ray diffraction data indicate that a considerable portion of coal substance, termed organized fraction, consists of large molecules arranged as two-dimensional sheets or platelets of condensed six-membered rings. Some of these sheets are stacked one upon the other. Other parts of the coal substance not arranged in sheets are considered to be unorganized.

The benzene-insoluble residue from coal hydrogenation has a diffraction pattern resembling that of coal; however, a serious problem encountered in examining this material is the interfering diffraction pattern of the ash, which unavoidably concentrates in the residue. Similarly, ash interferes with an interpretation of the pattern of natural coal. To correct these errors, considerable work has been done on ash-free model substances. Sugar chars appear particularly suitable for this purpose because they consist of comparatively simple, condensed colloidal systems containing no sulfur, nitrogen, or mineral matter. Furthermore, many chars react with hydrogen at high temperatures and pressures to yield asphaltene and oils similar to those formed in coal hydrogenation; thus they also may serve as models for hydrogenation studies.

A series of chars was made by heating sucrose in nitrogen for 1 hour at temperatures varying from 350° to 550° C. The yield of char decreased from 32 percent at 350° C. to 19 percent at 550° C. Over this temperature range the atomic ratios of hydrogen and oxygen to carbon decreased from 0.69 to 0.36 and from 0.21 to 0.083, respectively. These hydrogen-carbon ratios are similar to those of coals ranging in rank from bituminous to anthracite; however, the oxygen-carbon ratios of the chars are greater than for coals of similar hydrogen-carbon ratios.

The diffraction patterns of chars formed from sucrose, vanillin, kojic acid, rhamnose, conidendrine, and glycine were not greatly different from those of the coal vitrains previously studied. Identifiable bands show that the stack height and platelet diameters are approximately in the range for coals and indicate that molecular weights and packing are similar to those for coal.

X-ray diffraction also was applied, in studying the molecular structure of coal, to the distillate (melting point about 40° C.) from vacuum distillation (at 500° C.) of Pittsburgh-bed vitrinite; the diffractometer is shown in figure 2. The distillate contained planar layers similar to those of graphite as well as peripheral groups around the layers; moreover, some stacking of layers appears likely. These results show that the volatile matter in coals can be identified with other than the peripheral groups only and that the layers or platelets also may be distilled.

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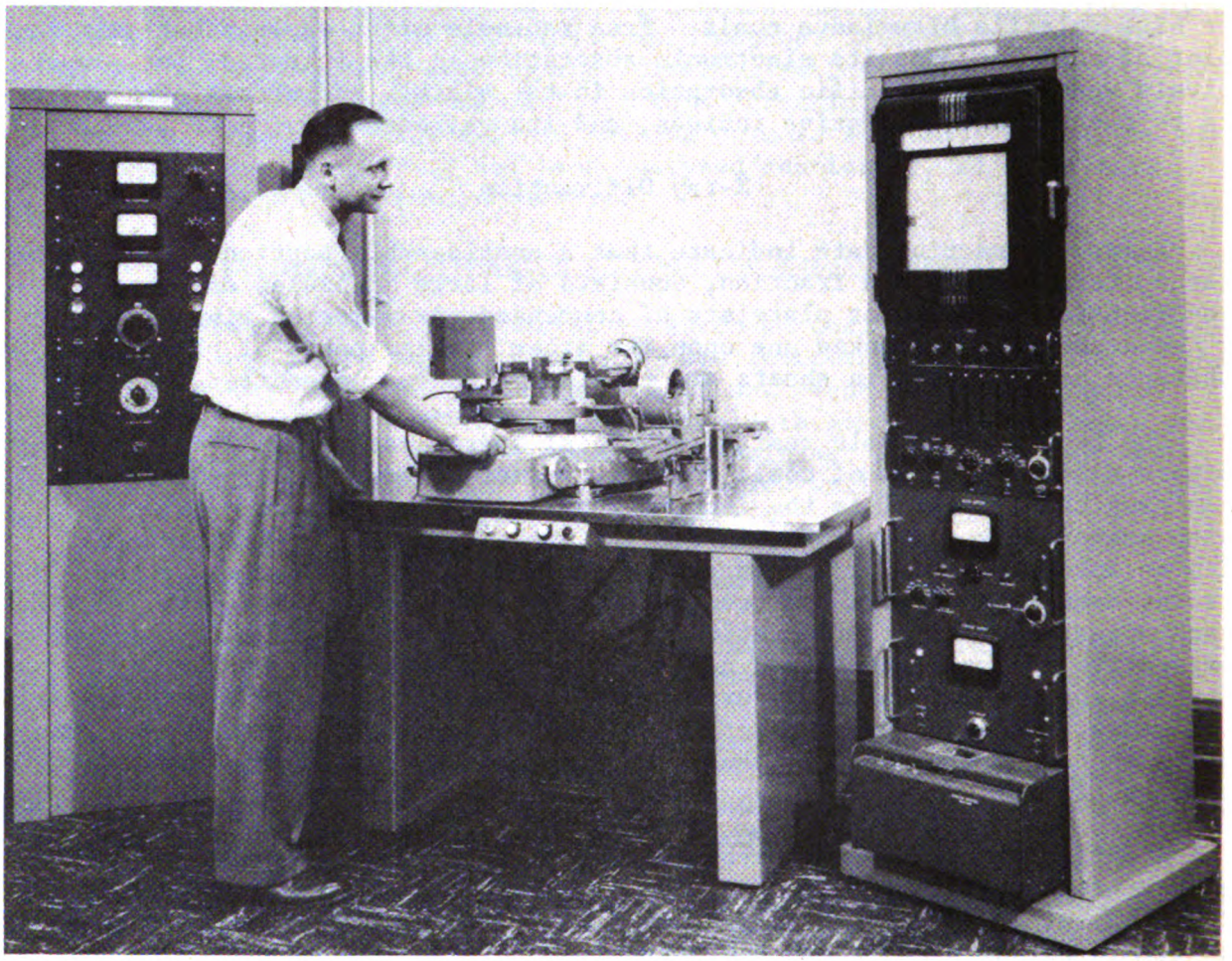

FIGURE 2. - X-ray Diffractometer.

Methods of evaluating X-ray data and the corrections hitherto applied for absorption of X-rays by a slab of coal have been reexamined. General equations for absorption corrections, applicable to poorly absorbing solids like coal, have been derived; and an experimental method of determining absorption coefficients has been developed. This determination is believed to be necessary for materials as heterogeneous as coal.

Pore Structure

Adsorption studies of American coals indicate that most of the pores in coal have openings of the order of molecular sizes (about 5 A. in the shortest dimension) and that many coals act as molecular sieves. For example, n-butane with a smallest cross-sectional diameter of 4.9 A. is adsorbed in very much greater quantities by most coals than isobutane, with the smallest diameter of about 5.9 A. The sorption of polar molecules appears to be a

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combination of physical adsorption and absorption, probably involving a mild chemical interaction.13/

Petrographic Studies

Since 1930, when the first practical laboratory method of microscopic evaluation and classification of coals was developed in the Bureau of Mines by the late Dr. Reinhardt Thiessen, more than 200 coals of varied rank, type, and source have been studied by this technique. Extensive information has been collected concerning the physical structure of the coal occurring in various beds in all the coalfields of the United States. A compilation of all the petrographic data obtained by the Bureau of Mines was published.14/ In addition to petrographic analyses this report includes chemical analyses, results of incidental tests and determinations, data from certain research studies of selected petrographic samples of coals, and geological and mine information on source of samples, with descriptive vertical sections of the coalbeds sampled. This information is supplemented with a general explanation of the Bureau of Mines method of examining coal in thin section and the system of describing and classifying it by type on the basis of microscopical analysis.

Investigation of the petrographic composition of lignite15/ and bituminous coals is being continued. Hernshaw coal of West Virginia, containing distinctive layers of bright and splint coal, was selected for the latter studies. Microscopic thin sections showed that the bright layer contained predominantly anthraxylon, while the splint layer contained mostly spores, translucent humic matter, and opaque attritus.

Bright coals generally make good coke, and splint coals poor coke. The agglutinating value of the splint coal (2.0) was in the range normally associated with noncoking coals, whereas that of the bright coal (6.8) was normal for coking coals. The free-swelling indexes, 1 and 7-1/2, respectively, also indicated the same distinction; however, coke from the splint layer was appreciably stronger than that from the bright coal. These results clearly indicate that neither simple petrographic analysis nor empirical tests are consistently valid criteria of coking behavior. Physical-chemical studies of petrographic components are needed to develop more consistent and sounder explanations and predictions of their behavior in utilization.

As part of long-range, fundamental studies of the nature of the petrographic components of coal, separation of macerals has been initiated. Of the four principal macerals, vitrinite and fusinite are easily obtained in sufficiently pure form in quantities desired in a laboratory. On the other hand, micrinite and exinite are distributed finely, and their separation presents some difficulty. A splint coal that contains all major macerals

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13/ Anderson, R. B., Hall, W. K., Lecky, J. A., and Stein, K. C. Sorption Studies on American Coals: Jour. Phys. Chem., vol. 60, No. 10, 1956, pp. 1548-1558.

14/ Parks, B. C., and O'Donnell, H. J., Petrography of American Coals: Bureau of Mines Bull. 550, 1956, 193 pp.

15/ Traverse, Alfred, Behavior of Petrographic Components of North Dakota Lignite in Preparation, Low-Temperature Carbonization, and Steam-Drying: Econ. Geol., vol. 50, No. 1, July 1955, p. 102.

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was selected for separation. By flotation, about 87 percent pure exinite and micrinite have been obtained. The following properties of macerals are being determined: Helium density, mercury density, surface areas by nitrogen and water-sorption isotherms, visible light-absorption coefficient, refractive index; diffraction characteristics by X-ray, electron, and neutron diffraction; chemical composition, and swelling properties.

Mild Chemical Treatment

Chemical reactions followed by infrared examination have been used to study the nature of the functional groups in coal. In most instances a method was tested first on polynuclear organic compounds and asphaltenes from coal hydrogenation, both of which may serve as models for coal.

Asphaltene has been found to react with maleic anhydride in a Diels-Alder addition, which indicates the presence of diene systems such as are found in anthracene. This reaction shows promise of introducing solubilizing groups into asphaltene and coal.

A new system, lithium in ethylenediamine, has been found for reducing aromatic rings, phenols, ketones, olefins, and acetylenes and for cleaving ethers at 100° to 110° C. It is probably the most powerful metal-amine-reducing system known. Infrared examination of treated anthraxylon and asphaltene showed extensive hydrogenation of aromatic rings and a large decrease in phenolic groups. Reduction of coal with lithium in ethylenediamine is being pursued.

The presence of phenols in coal has been demonstrated by several investigators and methods. These methods do not give consistent results, and all possess some degree of uncertainty in the presence of groupings other than hydroxyl. Recent Bureau work revealed that hexamethyldisilazane, $(CH_3)_3SiNHSi(CH_3)_3$, reacts not only with hydrogen-bonded phenols but also with highly hindered phenols that resist treatment with other reagents. The trimethylsilyl ethers formed are stable but can be readily hydrolyzed to the free phenols. The ethers have intense and distinctive infrared absorption bands. Infrared spectra of different coal samples, treated with hexamethyldisilazane to form trimethylsilyl ethers of the phenolic groups, indicate almost complete elimination of hydroxyl groups. The original hydroxyl content of the coal is determined by analyzing the treated coal for silicon. This method may be the most direct one for determining the true hydroxyl content of coals. Infrared spectra of the treated coals give little, if any, indication that coal contains hydrogen-bonded quinoid structures.

Homogeneous hydrogenation catalysts, which are not poisoned by sulfur, may catalyze the addition of hydrogen or synthesis gas to coal at relatively low temperatures. Thus coal reacts with carbon monoxide and hydrogen in the presence of dicobalt octacarbonyl at about 200° C. The extent of reaction and the products are greatly influenced by the solvent used. Benzene is a poor solvent, but the reacted coal shows indications of hydrogenation as well as introduction of carbonyl groups. The reaction proceeds further in ethanol, and the product shows greater solubility in the solvent. In addition, the

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reacted coal appears to have formed ethyl-ether groups. Phenol is the best solvent tested; 52 percent of reacted coal is soluble in phenol and 44 percent in pyridine.

To clarify the nature of the reaction of coal with synthesis gas in the presence of dicobalt octacarbonyl, polynuclear hydrocarbons were treated under similar conditions. The hydrocarbons generally were hydrogenated; reduction occurred at specific bonds in the molecule.

Microbial Degradation

Studies of bacteriostatic and fungistatic materials in coals have shown that the materials isolated are active against test cultures at dilutions of up to 1:20,000; a new assay method has been developed to test the potency of these materials. In certain instances the materials are present in the coals at a concentration up to 25 times the amount necessary for in vitro inhibition. Further purification of the materials may yield fractions of increasing potency, perhaps enough to enter the antibiotic range.

Slack lignites and weathered bituminous coal are more soluble in water than the usual coal samples, and cultures originally isolated from lignites grow better on slack lignites. Methods of increasing the growth on these materials, as well as increasing the degree of attack by microorganisms, are being developed.16/

In microbial attack of pure compounds with structures similar to portions of the coal molecule 3-hydroxy-2-naphthoic acid has been isolated as an intermediate during fermentation of the anthracene; this demonstrates an end-ring attack similar to that of phenanthrene, where 1-hydroxy-2-naphthoic acid is formed.

Indirect evidence indicates that two different types of enzymes are responsible for these transformations. For anthracene (and naphthalene), enzyme attachment and oxidation are assumed to occur on the same ring, but, for phenanthrene, attachment occurs at the 9:10 position, and oxidation, at an adjacent (end) ring. This postulate is confirmed by adaptation studies in which cells grown on naphthalene do not oxidize phenanthrene. If cells are grown on 2-methylnaphthalene, which possesses a bond structure similar to that of phenanthrene, oxidation of the phenanthrene does occur. Studies also have shown that the 9:10 bond of phenanthrene is involved in the end-ring oxidation, as substitution at that point inhibits the oxidation process.

Mathematical Treatment of Data

Data relating to numerous physicochemical phenomena can be presented as families of parallel straight lines - absorption, diffusion, crystallization, distillation, filtration, flame stability, heat transfer, hydroextraction, reaction kinetics - or lines having common intercepts - diffusion, reaction

16/ Rogoff, M., Golden, P. L., and Rosa, C., Inexpensive, Space-Saving Laboratory Shaker: Rev. Sci. Instr., vol. 27, No. 6, June 1956, p. 413.

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kinetics, hydrolysis, permeability, fluidization, adsorption, physical properties of solids and liquids. A method has been developed for treating such data statistically[17] by simultaneous least-square regression of all lines. It is simple and leads to a better estimate of values of the common or individual slopes and intercepts than can be obtained by separate least-square lines.

Composition of Perlite

Perlite is a rock that, when heated to a suitable temperature, expands to form a lightweight glasslike material with a cellular structure. Expanded perlite is used primarily as a lightweight aggregate, replacing sand and gravel in plaster and concrete. It is used also as an insulating, foundry, well-drilling, and filtering material, and as a filler material for paints, plastics, and fertilizers. Owing to the rapid growth of the perlite industry in recent years, the Bureau of Mines has received numerous requests for information about the possible harmful effects of breathing dust while processing and using perlite.

As published information on the composition of perlite is rather meager, samples of crude and expanded perlite from nine deposits in Arizona, California, Colorado, New Mexico, Nevada, and Utah were analyzed in cooperation with the Branch of Health Research.[18]

The chemical analyses of the perlite ore from the various sources were remarkably similar. The range of analysis was as follows: Moisture determined at 105° C., 0.1 to 0.6; loss on ignition at 800° C. (less moisture), 3.3 to 4.9; silica (SiO_2), 71.2 to 74.4; alumina (Al_2O_3), 12.4 to 14.0; ferric oxide (Fe_2O_3), 0.5 to 1.5; titania (TiO_2), 0.03 to 0.21; calcium oxide (CaO), 0.5 to 1.0; magnesia (MgO), 0.1 to 0.3; sodium oxide (Na_2O), 2.9 to 4.1; potassium oxide (K_2O), 4.0 to 5.0; and, sulfur trioxide (SO_3), 0.0 to 0.2 percent. Presumably the loss on ignition is due to evolution of the entrapped water.

EXPLORATION AND MINING

Exploration of Alaskan Coals

The U.S. Departments of Defense and the Interior have cooperated in developing an Alaskan coal-mining industry that can be depended on to supply fuel to the principal defense installations and the greatest population centers. As Alaskan coal-bearing lands are public domain, the Department of the Interior has been concerned with geologic mapping of coal-bearing areas by the Federal Geological Survey and with diamond drilling by the Bureau of Mines to determine the position, size, and quality of the coalbeds. Nearly

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17/ Ergun, Sabri, Application of the Principle of Least Squares to Families of Straight Lines: Ind. Eng. Chem., vol. 48, No. 11, 1956, pp. 2063-2068.

18/ Anderson, F. G., Selvig, W. A., Baur, G. S., Colbassani, P. J., and Bank, Walter, Composition of Perlite: Bureau of Mines Rept. of Investigations 5199, 1956, 13 pp.

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all drilling by the Bureau of Mines has been in the Matanuska coalfield, the principal source of coal for the Anchorage area.

Fieldwork during 1956 was designed to explore the western part of the Wishbone Hill District of the Matanuska field (see fig. 3). Earlier work by the Bureau of Mines and the Geological Survey demonstrated that the coalbeds in the eastern part of the district occupy a strong syncline. 19/ The western coalbeds had been assumed to occupy a similar syncline, but these beds were observed only where the (assumed) north flank of the syncline cropped out along the banks of Moose Creek, in the shallow workings of several small mines along these outcrops, and where they had been intersected to depths of several hundred feet by earlier diamond drilling.

In 1956 an attempt was made to discover the bedrock position of contact between the Eska Conglomerate and the Wishbone Hill formation by means of relatively shallow churn-drill holes into bedrock within the zone that had been projected from diamond drilling. It was desired to locate the contact in each of the fault blocks between the Buffalo fault and the Premier fault and in at least two places in the block west of the Premier fault. From this information the coal formations should be identifiable and appraisable with a minimum of additional diamond drilling. Five churn-drill holes were completed, one was started but recessed because of drilling difficulties, and one was started but recessed because of freezing weather. These holes are shown on the accompanying plan (fig. 3) as MC-8 to MC-14. Each hole penetrated 50 to 160 feet of overburden and was continued far enough to permit tentative identification of the bedrock formation; 1,178 linear feet of churn drilling was completed.

The work provided nearly conclusive evidence that the coal-bearing horizons of the south flank of the syncline within the block between the Buffalo fault and the Baxter fault reach bedrock (below 50 to 160 feet of overburden) within a zone whose northern boundary is about 500 feet south of hole MC-9. The distance along the beds, from outcrop to outcrop, is approximately 6,000 to 7,000 feet. At the synclinal axis the coalbeds attain a depth of about 2,000 feet. Thus the apparent reserves of the Premier series in this block are increased by 800 to 900 percent; existence of the Jonesville series in this block had not been demonstrated previously. The work has not demonstrated fully yet that the Premier series continues to the south-flank outcrop, but the inference is strong; additional drilling will be required to prove this inference and to determine the actual position and nature of the coalbeds in the block between the Baxter fault and the Premier fault and in the block immediately west of the Premier fault.

Anthracite Mining

Research and engineering activities in anthracite mining aim to (1) improve the efficiency of mining operations and reduce mining costs; (2) enhance the safety of the miner and make his tasks less arduous; (3) increase

19/ Barnes, Farrell F., and Payne, Thomas G., The Wishbone Hill District, Matanuska Coal Field, Alaska: Geol. Survey Bull. 1016, 1956, 88 pp.

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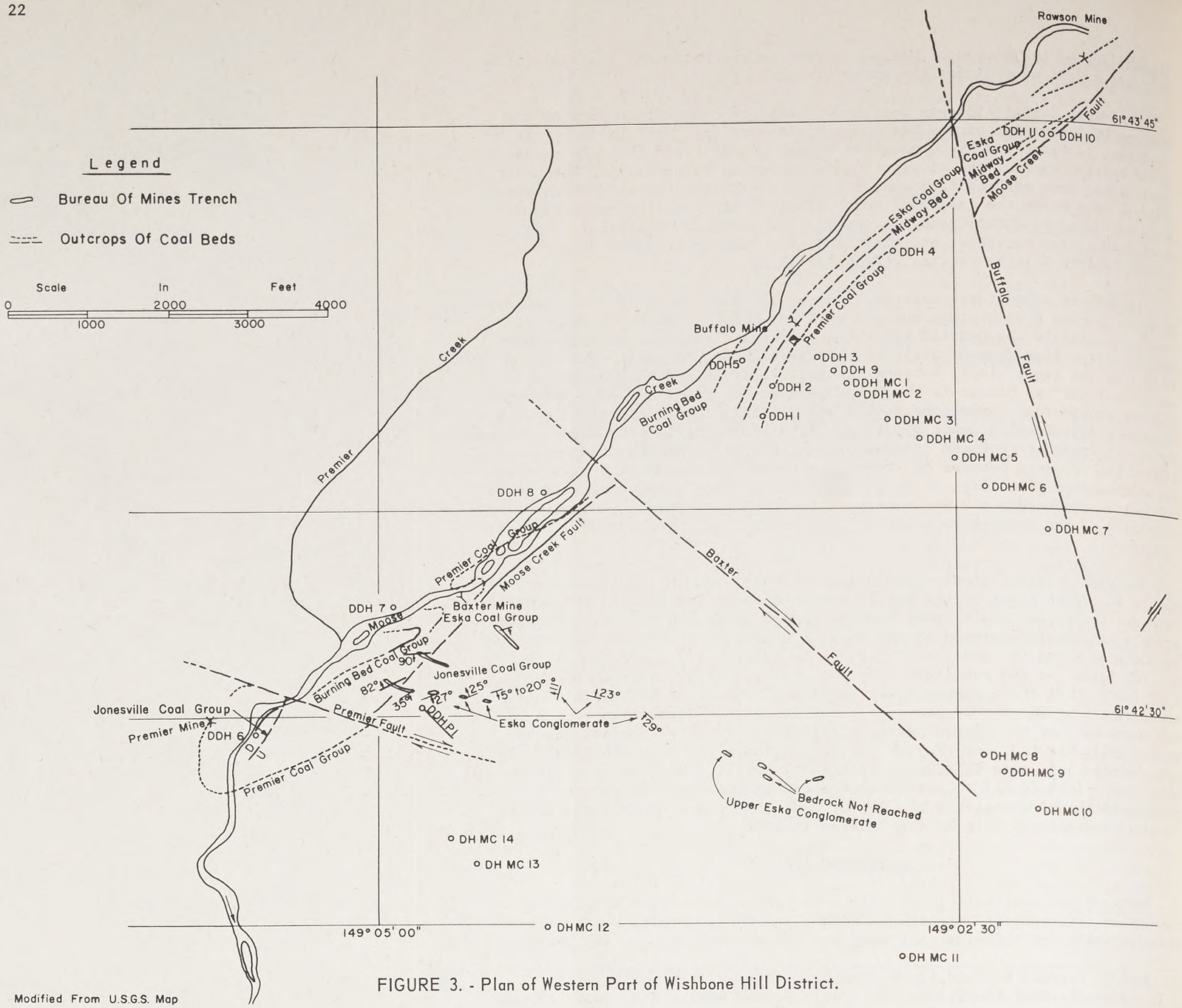

Modified From U.S.G.S. Map

FIGURE 3. - Plan of Western Part of Wishbone Hill District.

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recovery; and (4) contribute to mining technology by collecting, analyzing, and disseminating technical and scientific data obtained during the course of experiments.

Results of experiments with yielding-steel props have been published.20/

Pneumatic Coal Planer

High-speed mining with heavy equipment, including continuous miners, universal cutters, loading machines, and shuttle cars, has not proved entirely satisfactory on pitches exceeding 10°. A productive, safe, and efficient method is needed for mining beds pitching 10° to 25°, and modern longwall mining with planing equipment may provide the answer.

In 1955 underground tests of the coal planer established that hard anthracite can be planed. Efforts are now directed toward perfecting a longwall method for anthracite mines (see fig. 4).

A section was selected from test sites offered by operating companies; the coalbed is 8 feet thick, 1,200 feet long, 400 feet wide, and pitches 12° to 18°. The thickness is somewhat greater than desired for trouble-free planer operation, but the roof and floor were the most suitable for testing purposes.

Scraper-Shaker Loader

A location has been found in the Western Middle Field where the loader will be used to drive a 30° rock slope in opening a new section of a mine. If underground conditions are favorable, the machine will be taken inside the mine for gangway development. The location for a new mine opening that would best suit a layout for systematic longwall mining was chosen after correlating data available on existing mine maps of the two minable coalbeds in the area. The bed selected is about 5 feet thick, and the pitch is expected to vary between 5° and 12°.

Pneumatic Packing Machine

When cleaning-plant refuse was used for packing (2-inch top size), the Brieden pneumatic packing machine performed best with an average mixture, that is, when each of the four sizes screened as test samples approached 25 percent.21/ Excessive fines resulted in pipeline blockages. The packing rate was approximately 1,000 cu. ft. per hour.

As work progressed, the length of the discharge line increased from 260 to 1,220 feet. The amount of material handled remained fairly constant for

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20/ Hartley, J. C., Cooner, J. D., Sr., and Brennan, R. J., Anthracite Mechanical-Mining Investigations. Use of Yielding Steel Supports (Props) in Combination With Backfilling for Mining Thick, Flat Beds: Bureau of Mines Rept. of Investigations 5290, 1956, 29 pp.

21/ Whaite, R. H., Anthracite Mechanical Mining Investigations. Second Testing of Brieden Pneumatic Packing Machine: Bureau of Mines Rept. of Investigations 5273, 1956, 22 pp.

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distances up to 600 feet; from 600 to 900 feet capacity decreased about 8 percent; and for distances over 1,000 feet the loss was almost 50 percent. These data are not conclusive, because the air pressure available at the machine varied from 60 to 67 percent of the manufacturer's rated requirement. Air requirements were about 2,400 c.f.m. An average volume of 139 cu. ft. of air was consumed in packing 1 cu. ft. of material.

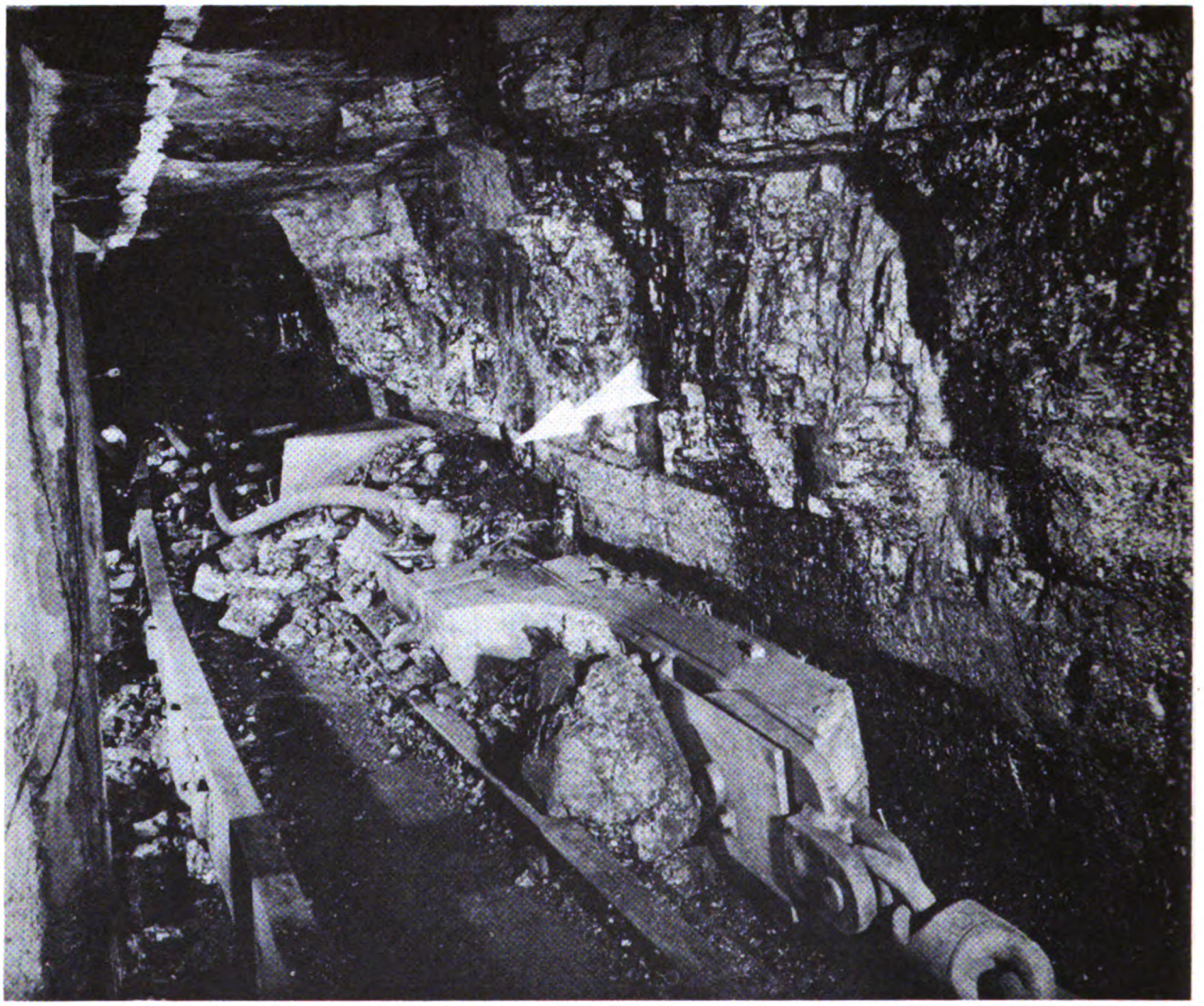

FIGURE 4. - Bureau of Mines Experimental Pneumatic Coal Planer. Arrow points to cutting edge.

On the basis of data obtained in this and one previous experiment, pneumatic packing offers a means of supporting strata in mining. The problem is largely one of adapting this method of backfilling to the daily cycle of mining operations. The capacity is great enough for a single packing machine to fill voids, including chamber openings, in a pillar-robbing operation producing a total of 240 tons in two shifts per 24-hour day; packing is done on the

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third shift. In fact, the packing rate required was only 63 percent of the rated capacity of the packing machine. The cost of labor (1955 base) is estimated at 35 cents per cubic yard of material packed, and the cost of compressed air, 19 cents.

Mine-Drainage Control

Anthracite mine-drainage activities included a study of the general anthracite-mine water problem and an engineering evaluation of individual programs or projects to assure that they are planned, designed, and executed in compliance with the spirit and specific requirements of Public Law 162, 84th Congress. This law authorizes the Secretary of the Interior to contribute an amount not to exceed $8,500,000 to the Commonwealth of Pennsylvania, matched by Commonwealth funds, for control and drainage of water, which, if not so controlled or drained, will flood anthracite formations. This program is in line with the policy to conserve a valuable natural resource, promote national security, prevent injury and loss of life, and preserve public and private property.

In 1956 two water-control projects were approved; both were deep-well pump installations to control water pools in abandoned mines to prevent inundation of adjoining active mines. Approval of these projects by the Secretary of the Interior was preceded by a detailed review of the projects, including comprehensive studies of rainfall, runoff, infiltration, pumping records, geologic structures, and mine workings; all were examined with the cooperation of the Federal Geological Survey. Final evaluation of each project included endorsement of the plan and appraisals of specifications, costs, and construction methods of the facilities to be provided.

Work on five additional projects had progressed nearly to submittal for final approval. One project is stream-bed improvement to reduce seepage of surface water into active and abandoned mine workings, and the other four are deep-well pumping plants, with a total capacity of 42,000 gallons per minute. Additional pumping and surface-improvement projects are in various stages of completion before being submitted for approval. Analytical data are being collected for four deep-well pumping plants, eight stream-bed and surface-improvement projects, and a number of projects that require backfilling of old strip-mining pits through which large volumes of water enter the mines.

Bituminous Coal Mining

Mining projects were conducted in cooperation with mining companies to improve methods of mining, to increase production per man-shift and thereby reduce costs, to increase the percentage recovery of coal for conservation of coal resources, and to attain each of these objectives with increased safety.

Augers for Surface Mining

Auger mining of coal from highwalls of active strip mines, from reopened strip mines, and from benched outcrops has increased rapidly since World War

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II; in 1955 production by this method was estimated at 6 million tons. From the standpoint of conservation, auger mining is used to best advantage in recovering coal between abandoned underground mines, adjacent strip mines, and in areas where mining by other methods is not feasible for reasons such as extremely weak roof, where a thick overlying sandstone would necessitate difficult drilling and expensive blasting in stripping, or where coal would have to be stripped selectively to obtain a salable product.

Mining methods with coal augers were studied in eight operations in five coalbeds in three States. Diameters of the auger holes ranged from 26 to 52 inches; production per shift, 65 to 503 tons; and estimated recovery, 20 to nearly 50 percent. Figure 5 shows 48-inch holes drilled in the highwall of a strip mine.

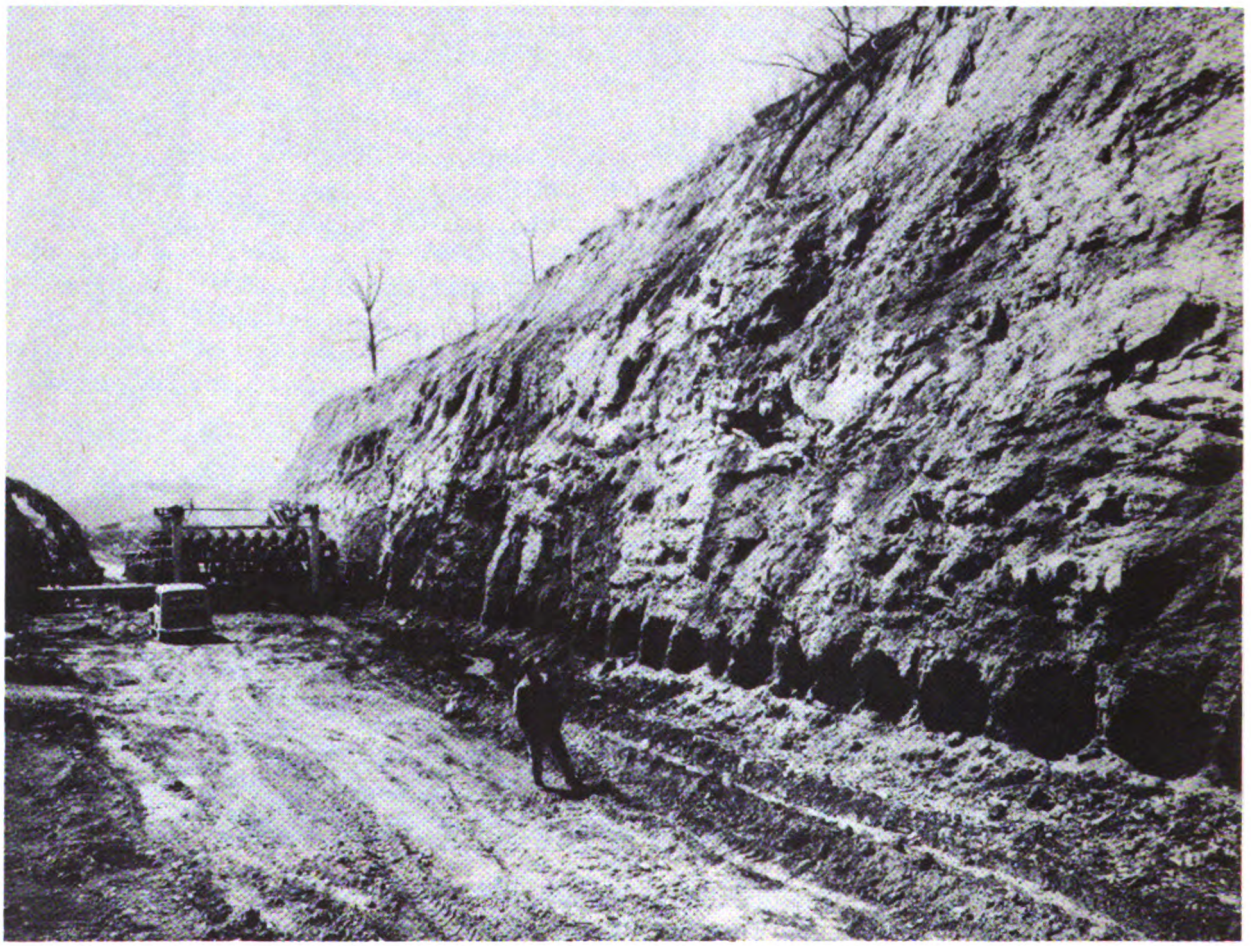

FIGURE 5. - Highwall With 48-Inch Auger Holes.

Longwall Mining

Four coal planers produced an estimated 640,000 tons of raw coal during the year. Fieldwork in connection with longwall mining was completed, and a

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report is being prepared. To July 1956 over 1-1/2 million tons of raw coal had been mined with planers. Recovery at four mines was 83, 71, 78, and 83 percent as compared with 78, 53, 59, and 55 percent by conventional methods at the respective mines.

Roof supports on the longwall faces were: GHH props, shop-made steel bars, and wood cribs with crib releases at Stotesbury No. 8 mine in southern West Virginia; yielding-type Uerdinger props, GHH bars, and wood cribs with releases at Pine No. 1 mine in central Pennsylvania; Gerlach props, GHH bars, and wood cribs with releases at Amherst No. 4 mine in southern West Virginia; and wood props with cribs at Quality No. 2 mine in western Arkansas.

Methods and Equipment for Underground Development

Fieldwork connected with the study of methods and types of equipment used in underground development of bituminous coal mines was completed during the year, and a report is being prepared. The study included mines operated in coalbeds from 35 to 84 inches thick in Pennsylvania, Ohio, West Virginia, and Kentucky; trackless and track-mounted mobile loading, continuous-mining, and hand-loading methods were used.

In the past, when development entries normally were narrow, the output per man for mine development usually was less than the production rate from panels. With mechanized equipment, panels are mined out more rapidly. As the type of equipment usually used for development is the same as that used for production work, development now is more rapid, and rates of production markedly increased. A study of development practices in 14 mines reveals that output per man now approaches and in some mines equals that obtained in regular production. Likewise, the advance in sets of development entries has improved and ranges from 5 to 22 feet per shift for each entry in the set.

Costs of Timber and Roof-bolt Supports

The additional cost of roof bolting over that of timbering may be offset by other advantages that contribute to a more efficient operation. A study was made to compare mining by the two methods under similar mining conditions.

Detailed studies were made during the year at Mine 17, Red Jacket Division, Island Creek Coal Co.; Kopperston No. 2 mine, Eastern Gas and Fuel Associates; Olga No. 2 mine, Olga Coal Co.; and No. 2 mine, Boone County Coal Corp.

Field data have not been correlated and evaluated fully, but roof bolting apparently is not solely responsible for the recent increases in rates of coal production, although it is a contributing factor. Improvements in mining methods, equipment, and management each have contributed to more efficient operation.

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Systems and Equipment for Face Haulage

At many mechanized bituminous coal mines, especially where continuous-mining machines are used, coal is said to be mined faster than it can be transported from the face. Face transportation is being studied to collect and correlate data as a basis for selecting plans and equipment most suitable under given conditions.

Fieldwork was begun at five mines using the following equipment.

Mine No.	Loading equipment	Face haulage equipment
1.....	Mobile loading, continuous mining.	Shuttle cars, extensible belts.
2.....	Continuous mining.	Extensible belt.
3.....	Track loading.	Mine cars.
4.....	Continuous mining.	Extensible belts, shuttle cars.
5.....	Continuous mining.	Chain conveyors with bridge conveyors.

Recovery of Coal

The Bureau of Mines often is asked to estimate percentages of recovery of coal. Reliable figures for a particular bed are available now only for small areas. Virtually all older estimates have been proved too high and do not show the actual loss of resources caused by low recoveries.

A study of this problem has been started in the Upper Freeport bed, covering a determination of recovery for each method of mining in that bed. Preliminary work consists of planimetering maps and computing recovery four times in the "Thick Freeport" areas, with and without extraction of pillars. One difficulty encountered thus far is the inability to obtain complete production records for mines, because records are lost, and ownership is transferred frequently.

Reserves of Coking Coal

During 1956, 13 reports were published on coking-coal reserves in counties where fieldwork had been completed before June 30, 1955. Forty-four reports have been published to complete the project. Figure 6 shows the counties for which reports have been completed in Pennsylvania, West Virginia, Kentucky, and Maryland; figure 7, for Tennessee.

Marshall County, W. Va.

The Pittsburgh bed is the important one in this county, and nearly all production to date has been from it.[22/] The remaining volume of reserves in

22/ Dowd, J. J., Provost, J. M., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Marshall County, W. Va.: Bureau of Mines Rept. of Investigations 5207, 1956, 21 pp.

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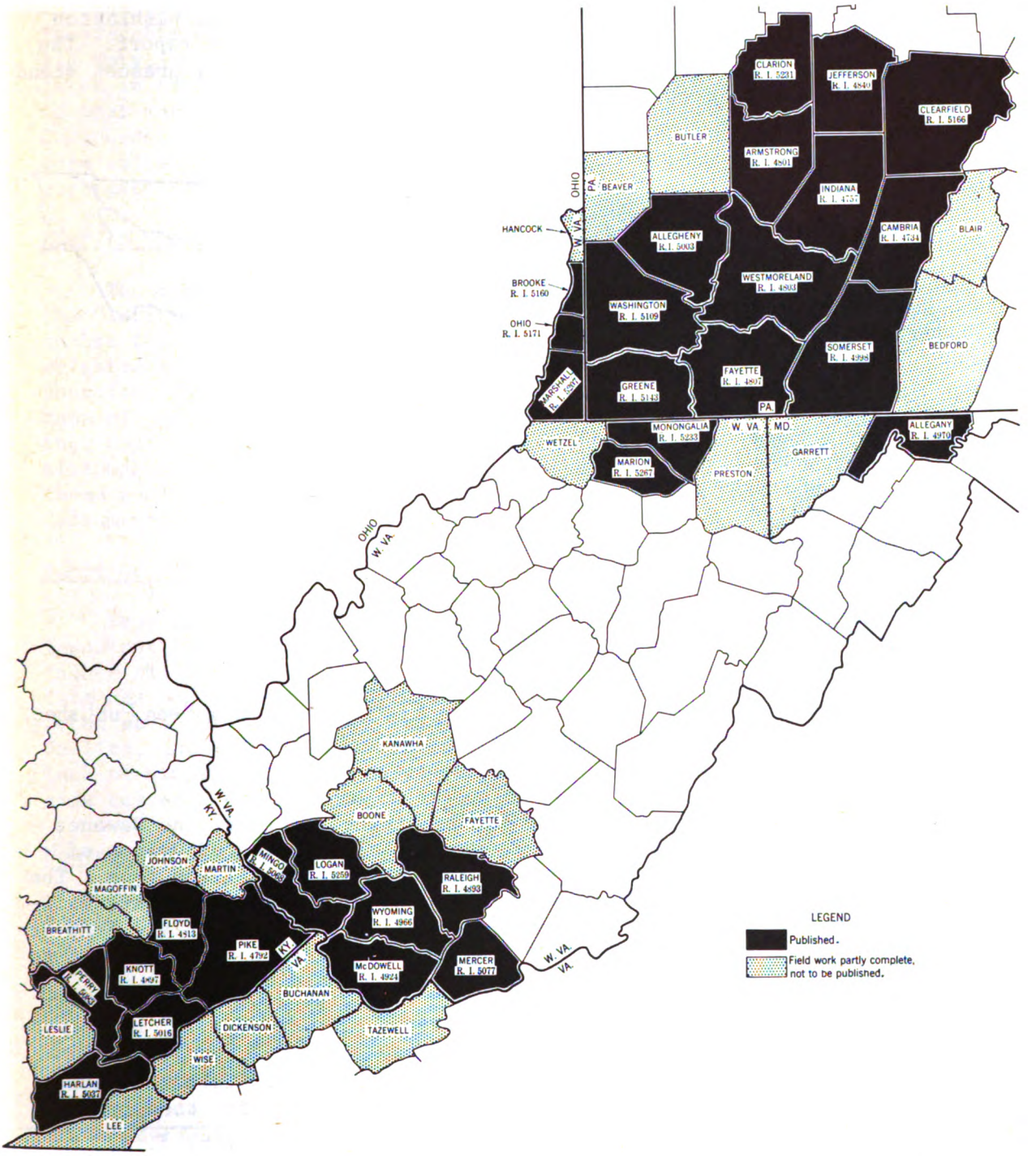

FIGURE 6. - Counties in Pennsylvania, West Virginia, Kentucky, and Maryland for Which Estimates of Coking Coal Reserves Have Been Published.

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all beds 28 inches and more thick is estimated at 3,071 million tons. Recovery assumed at 46.1 percent is 1,416 million tons. Maps of the Washington, Waynesburg, Sewickley, and Pittsburgh beds are included in the report. The sulfur content in Pittsburgh coal in this county is too high by present standards for manufacturing good metallurgical coke.

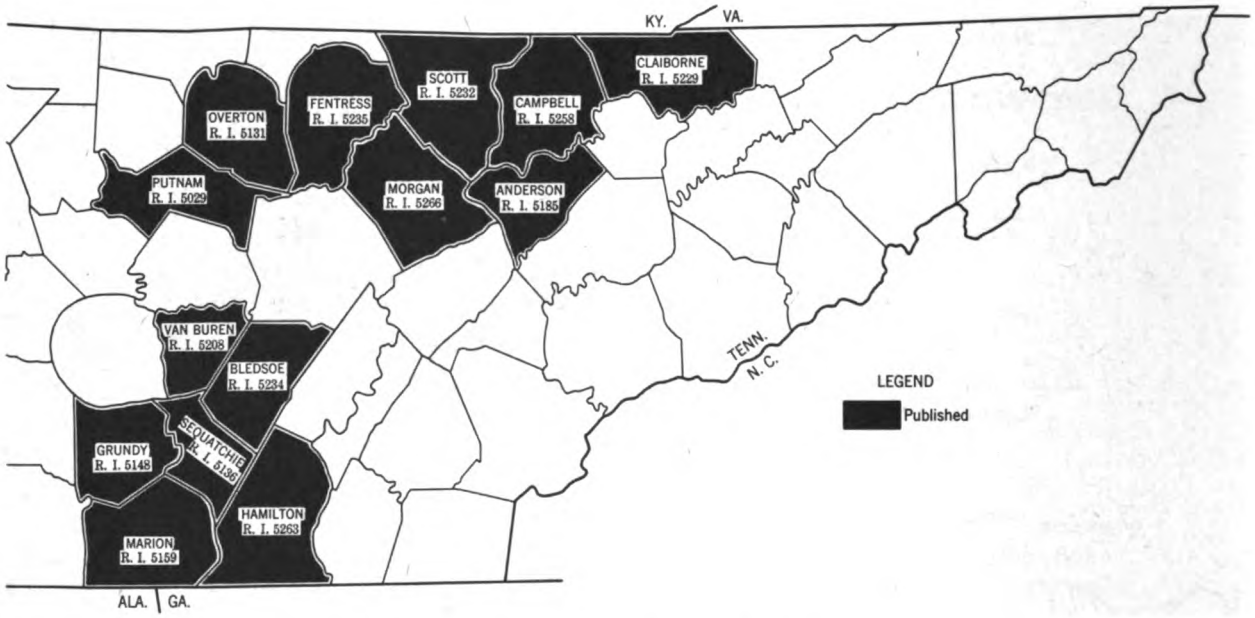

FIGURE 7. - Counties in Tennessee for Which Coking Coal Estimates Have Been Published.

Van Buren County, Tenn.

From the standpoint of production and remaining reserves, the Sewanee bed is the most important one in this county.23/ The estimated volume of reserves in three coalbeds 28 inches and more thick is 15 million tons. The average recovery of coal is 50 percent; hence recoverable reserves are estimated at 8 million tons. Maps of the Lantana, Sewanee, and Richland beds are in the report. Although one sample of Sewanee coal could not be prepared to make metallurgical coke, most of the coal can be cleaned suitably.

Claiborne County, Tenn.

The Coal Creek and Jellico beds are the important ones from the standpoints of production and reserves.24/ An estimated 55 million tons remains

23/ Hershey, R. E., Williams, Lloyd, Crentz, W. L., and Miller, J. W., Estimate of Known Recoverable Reserves and Preparation Characteristics of Coking Coal in Van Buren County, Tenn.: Bureau of Mines Rept. of Investigations 5208, 1956, 25 pp.

24/ Hershey, R. E., Williams, Lloyd, Crentz, W. L., Miller, J. W., and Reynolds, D. A., Estimate of Known Recoverable Reserves and Preparation and Carbonizing Properties of Coking Coal in Claiborne County, Tenn.: Bureau of Mines Rept. of Investigations 5229, 1956, 44 pp.

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in beds at least 28 inches thick. Based upon 57.7 percent average recovery, recoverable reserves are estimated at 32 million tons. Maps of the Pewee, Big Mary, Lower Pioneer, Jellico, Blue Gem, Coal Creek, and Rex beds are given in the report. Pewee, Jellico, and Coal Creek Coals can be prepared to meet current requirements for metallurgical coal. One sample each of Coal Creek and Jellico coal was carbonized; Coal Creek coal was shown to be more suitable for metallurgical use than Jellico coal because the coke is stronger and contains less sulfur.

Clarion County, Pa.

The largest production comes from the Lower Kittanning bed, although the Clarion bed has the largest volume of reserve.25/ In beds at least 28 inches thick, the volume of reserves is estimated at 790 million tons. The weighted-average recovery for all coal in this county has been 54.2 percent; therefore the volume of recoverable reserves is estimated at 428 million tons. Maps of the Upper Freeport, Lower Freeport, Middle Kittanning, Lower Kittanning, Upper Clarion, Lower Clarion, and Brookville beds are in the report. Although Lower Kittanning coal contracts during carbonization, it yields well-fused coke. For metallurgical purposes it should be blended with low- or medium-volatile coal.

Scott County, Tenn.

From the standpoint of production, the Poplar Creek bed is the most important in this county.26/ The volume of reserves in beds more than 28 inches thick is 74 million tons. The average weighted recovery of coal to date has been 52.4 percent, hence the volume of recoverable reserves is estimated at 39 million tons. Maps of the Pewee, Red Ash, Big Mary, Windrock, Jellico, and Poplar Creek beds are included in the report. Because of the lack of sampling places, only Poplar Creek and Big Mary coals were float-and-sink tested. Poplar Creek coal can be upgraded for making metallurgical coke.

Monongalia County, W. Va.

The Pittsburgh, Sewickley, and Upper Freeport beds are the most important in the county at this time.27/ The volume of reserves in all beds at least 28 inches thick is estimated at 3,294 million tons. Based upon a weighted-average recovery of 47.9 percent, the volume of recoverable reserves is estimated at 1,577 million tons. Maps of the Washington, Waynesburg, Sewickley, Redstone, Pittsburgh, and Upper Freeport beds are in the report. Pittsburgh and Upper Freeport coals can be used for making metallurgical coke, the former especially when blended with low-volatile coals.

25/ Blaylock, D. W., Dowd, J. J., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Clarion County, Pa.: Bureau of Mines Rept. of Investigations 5231, 1956, 27 pp.

26/ Williams, Lloyd, Gibbs, H. K., Crentz, W. L., and Miller, J. W., Estimate of Known Recoverable Reserves and Preparation Characteristics of Coking Coal in Scott County, Tenn.: Bureau of Mines Rept. of Investigations 5232, 1956, 36 pp.

27/ Travis, R. G., Dowd, J. J., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Monongalia County, W. Va.: Bureau of Mines Rept. of Investigations 5233, 1956, 30 pp.

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Bledsoe County, Tenn.

The Morgan Springs, Lantana, and Sewanee coalbeds have been mined on a very limited scale.28/ Only 22 million tons is estimated to remain in beds at least 28 inches thick. Based upon an estimated recovery of 50 percent, the volume of recoverable reserves is 11 million tons. Maps of the Morgan Springs, Lantana, and Sewanee beds are included in the report. Because of the limited reserves and moderate mining in the county, no coal was sampled.

Fentress County, Tenn.

The Wilder bed is the most important from a production standpoint and also contains the largest volume of reserves.29/ About 66 million tons is estimated to remain in beds at least 28 inches thick. Based upon an average recovery of 62.8 percent, recoverable reserves are estimated at 41 million tons. Maps of the Nemo, Sewanee, Wilder, and White Oak beds are in the report. None of the coal tested could be upgraded to conform with present standards for metallurgical coal.

Campbell County, Tenn.

The Coal Creek bed is the most important one in the county.30/ Some 267 million tons of reserves is estimated to remain in beds at least 28 inches thick. The weighted average recovery from all beds was estimated at 55.9 percent, or 149 million tons of recoverable reserves. Maps of the Rock Spring, Petree, Pewee, Walnut Mountain, Red Ash, Big Mary, Windrock, Upper Pioneer, Jordan, Joyner, Jellico, Blue Gem, Coal Creek, and Poplar Creek beds are included in the report. Float-and-sink tests indicate that the Petree, Pewee, Walnut Mountain, Red Ash, Jordan, and Coal Creek coals, and Jellico coal in the eastern part of the county, are potential sources of metallurgical coal. Carbonizing tests indicate that Coal Creek coal is metallurgical grade; but Pewee, Windrock, and Red Ash coals probably are suitable only as minor constituents of coking blends.

Logan County, W. Va.

The Cedar Grove and Chilton coals are the most important from a production standpoint.31/ In beds at least 28 inches thick the volume of reserves

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28/ Williams, Lloyd, and Hershey, R. E., Estimate of Known Recoverable Reserves of Coking Coal in Bledsoe County, Tenn.: Bureau of Mines Rept. of Investigations 5234, 1956, 18 pp.

29/ Lowe, R. W., Crentz, W. L., and Miller, J. W., Estimate of Known Recoverable Reserves and Preparation Characteristics of Coking Coal in Fentress County, Tenn.: Bureau of Mines Rept. of Investigations 5235, 1956, 34 pp.

30/ Williams, Lloyd, Gibbs, H. K., Crentz, W. L., Miller, J. W., and Reynolds, D. A., Estimate of Known Recoverable Reserves and Preparation and Carbonizing Properties of Coking Coal in Campbell County, Tenn.: Bureau of Mines Rept. of Investigations 5258, 1956, 78 pp.

31/ Tavenner, W. H., Dowd, J. J., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Logan County, W. Va.: Bureau of Mines Rept. of Investigations 5259, 1956, 50 pp.

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is estimated at 3,939 million tons. The weighted average recovery for all beds is 60.7 percent, which leaves 2,391 million tons of recoverable reserves. Maps of the No. 5 Block, Stockton, Coalburg, Buffalo Creek, Winifrede, Chilton A, Chilton, Hernshaw, Williamson, Upper Cedar Grove, Upper Split of Cedar Grove, Cedar Grove, Lower Cedar Grove, Alma, and No. 2 Gas beds are included in the report. Virtually all coals in the county are coking coals and most are either of metallurgical grade as mined or can be prepared to meet present specifications for metallurgical coal.

Hamilton County, Tenn.

The important bed from the standpoint of production is the Richland, but the Sewanee has the largest volume of reserves.32/ About 35 million tons remains in beds at least 28 inches thick. The average recovery for all beds is 50 percent, and on this basis 17 million tons is recoverable. Maps of the Lantana, Sewanee, and Richland beds are included in the report. Coal from the Richland bed probably could be cleaned to produce a satisfactory metallurgical coal.

Morgan County, Tenn.

In this county the Poplar Creek bed is the most important.33/ About 59 million tons of coal is estimated to remain in beds at least 28 inches thick. Based upon an average recovery of 51.1 percent from all beds, the volume of recoverable reserves is estimated at 30 million tons. Maps of the Pewee, Red Ash, Big Mary, Joyner, Jellico, Blue Gem, Poplar Creek, Hooper, and Christmas beds are included in the report. Coal from the Pewee, Red Ash, and Big Mary beds were float-and-sink tested and can be upgraded by mechanical cleaning to yield a satisfactory metallurgical fuel.

Marion County, W. Va.

The Pittsburgh bed is the most important in the county.34/ The volume of reserves in beds over 28 inches thick is estimated at 2,983 million tons. The weighted-average recovery for all beds is 52.9 percent, hence the volume of recoverable reserves is 1,579 million tons. Maps of the Washington, Waynesburg, Sewickley, Redstone, Pittsburgh, Upper Freeport, Upper Kittanning, and Lower Kittanning beds are in the report.

32/ Hershey, R. E., Williams, Lloyd, Crentz, W. L., and Miller, J. W., Estimate of Known Recoverable Reserves and the Preparation Characteristics of Coking Coal in Hamilton County, Tenn.: Bureau of Mines Rept. of Investigations 5263, 1956, 27 pp.

33/ Williams, Lloyd, James, Curtis, Crentz, W. L., and Miller, J. W., Estimate of Known Recoverable Reserves and the Preparation Characteristics of Coal in Morgan County, Tenn.: Bureau of Mines Rept. of Investigations 5266, 1956, 43 pp.

34/ Provost, J. M., Dowd, J. J., Abernethy, R. F., and Reynolds, D. A. Estimate of Known Recoverable Reserves of Coking Coal in Marion County, W. Va.: Bureau of Mines Rept. of Investigations 5267, 1956, 30 pp.

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Foreign Activities

During 1956 the Bureau of Mines engaged in three coal projects outside the United States. As in previous years ICA made funds available for this work. The general objectives under such project agreements are to promote better safety and health conditions for mine workers and to increase production and utilization of solid fuels in underdeveloped areas.

Technical assistance was given to the Governments of Afghanistan, Colombia, and India. These projects remained active at the close of the year. Similar assistance programs in Nepal, Pakistan, and the Philippines were terminated during 1955. Further studies were made by an independent engineer for developing the Markarwal coal mines of Pakistan.

Afghanistan

Two engineers from the Federal Bureau of Mines were stationed in Afghanistan as technical advisors to the Royal Afghan Ministry of Mines for developing and operating the Karkar and Ishpushta coal mines and developing a potential coal-producing area at Darra-i-Suf. Coal production from these two mines was less than 20,000 tons in 1956. Although the local market for solid fuels is growing, difficult mining conditions, lack of suitable mining equipment, and limited electric power have restricted increases of production. The subbituminous coal is very friable, and degradation during handling from mine face to consumers in Kabul creates problems of marketing and utilization. A shortage of trucks for transporting coal and poorly maintained roads are also marketing problems.

The Afghan Ministry of Mines was assisted in selecting two briquetting machines purchased from a Swiss manufacturer. Briquets will be produced from the fine coal by using a locally available binder of sawdust, cannery wastes, or molasses. Assistance was continued in the layout and purchase of equipment for a new powerline to supply the Karkar mine from a hydroelectric station at Pul-i-Khumri. Recommendations were made for procuring automotive equipment (later partly supplied by ICA) and roadbuilding equipment, primarily for constructing an access road to the new Darri-i-Suf coal area. ICA ordered this equipment.

Colombia

During the year the Columbian Government corporation (Instituto de Fomento Industrial) concerned with industrial development completed a coal-preparation plant in the vicinity of Cali in the Cauca Valley. By the end of the year the plant was being tested, and its need for coal was increasing. The mines in the Cauca Valley that are to supply the plant have not yet attained an adequate rate of production, and the Bureau was requested by ICA to assign an engineer to continue mine development.

After resuming the project, the chief obstacles to increased production were lack of ventilation and other unsafe working conditions. Several fatal accidents occurred, and generally gassy conditions were extremely hazardous.

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Safety-testing equipment was obtained through the Bureau, and a strong safety campaign was started immediately.

Two additional needs for effective Bureau cooperation are: (1) Assignment of Colombian engineers to receive practical training by working directly with the Bureau engineer - Colombian authorities have assured their cooperation in this respect - and (2) adequate safety regulations. It is hoped that they are established before Bureau participation is withdrawn.

India

Bureau assistance in developing and utilizing the lignite deposits at South Arcot, Madras State, continued during the year, although no personnel was assigned permanently to the site. Early in the year two coal-utilization specialists who spent approximately 2 months in India, recommended that a pilot plant, using the Bureau-developed Parry drying process, be constructed with a capacity of 4,000 tons of raw lignite per year. Fine lignite from the dryer would be briquetted and the briquets carbonized. The tars would be investigated locally and at the Fuels Research Institute to determine their most advantageous use. Raw-lignite briquets would be available to the Madras powerplant and to railroads, and carbonized briquets would be used as domestic fuel.

ICA requested the Bureau to supervise engineering of the pilot plant and to assist in its installation. An allotment of $433,000 was made to the Bureau to purchase plant equipment and engineering services and to cover Bureau costs of additional test work, laboratory equipment, and administration. The design and contracting for equipment were well advanced in December, when unfortunately one of the Bureau experts, Mr. V. F. Parry, died; however, the work continued without interruption.

Health and Safety

One of the main purposes of the Bureau of Mines is to aid the mineral industries by determining causes of accidents and injuries, finding ways to prevent them, and imparting the knowledge to mine officials, workers, and safety men. The Bureau collects statistics and details on accidents from mines and plants; inspectors visit coal mines to observe conditions and investigate causes of accidents. New or improved safe practices are noted. When problems are beyond previous experience, they may require experimental study. The findings are not only published, but lectures, demonstrations, and papers are presented at safety meetings.

Roof Bolting

Methods have been developed for testing the effectiveness of anchorages obtained with various types of roof bolts. Data for 1-inch slotted and 3/4-inch expansion-type headed bolts indicate that for a given increment of load the displacement of expansion-type bolts was approximately three times that of slotted-type bolts in sandstone and twice that in shale.35/

35/ Barry, A. J., Panek, L. A., and McCormick, J. A., Anchorage Testing of Mine-Roof Bolts. Part 2. Expansion-Type, 3/4-Inch Bolts: Bureau of Mines Rept. of Investigations 5194, 1956, 19 pp.

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The upper limit of load that can be sustained by a bolt equals either its strength or its anchorage, whichever is smaller. From a safety standpoint, until installed roof bolts have been observed over long periods and under a variety of conditions, the strength of the bolt should be considered equal to the yield load. The yield load therefore should not be exceeded when bolts are tightened. Many 3/4-inch mild-steel (SAE C-1020) headed bolts yield at torques greater than 200 ft.-lb. (approximately 8,000 pounds load), owing to tensile and torsional stresses induced when the bolt is tightened.

To insure good roof control, roof bolts should be installed immediately after new roof is exposed; they should be preloaded enough to minimize sag of the roof over the opening. The problem of measuring preloading stress on a bolt was partly solved by using a torque wrench. Tests on slotted-type roof bolts, installed in a conventional manner, indicate that a torque of 260 ft.-lb. applied to the nut will produce a load of 10,000 ± 2,700 pounds on the bolt. Despite errors introduced by frictional effects in the nut-bolt-bearing plate assembly, the torque wrench is a practical instrument for checking the tension load of slotted-type roof bolts.

To determine the torque-load relationship for 5/8-inch-(nominal) diameter roof bolts, five makes of expansion shells and two types of bolts (regular and high strength) were tested in two sandstone and two shale mine roofs.36/ Unused flat-steel bearing plates in as-received condition were used. The relationship between torque and load was approximately 50 pounds of load per foot-pound of torque for both regular and high-strength bolts. This relationship is valid up to 130 ft.-lb. for regular bolts and 175 ft.-lb. for high-strength bolts. A torque of 130 ft.-lb. on a regular bolt will produce a load of 6,150 ± 1,910 pounds 90 percent of the time, and a torque of 175 ft.-lb. on a high-strength bolt will produce 8,050 ± 2,750 pounds 90 percent of the time. Above these values, the torque-load relationship tends to be erratic owing to binding between the bolt and plug threads. Binding expends excessive torque by producing torsion in the bolt rather than tension. This torsion adversely affects the yield load of the bolt; therefore the installed torques should not exceed the 130- and 175-ft.-lb. values.

Roof bolts have been installed largely on the basis of methods determined by experimentation before general use throughout the mine. Very little work had been done to develop mathematical formulas that could be used to determine spacing of bolts, effective angle of installation, best anchoring device, or best diameters of drill holes and rods.37/ Model testing as applied to roof bolting required theoretical and experimental stress analysis under centrifugal testing conditions. Dimensional analysis was used to derive a roof-bolting design formula in terms of the structural variables. This formula was transformed into an exact equation after testing the models.

36/ Barry, A. J., Panek, L. A., and McCormick, J. A., Use of Torque Wrench to Determine Load in Roof Bolts. Part 3. Expansion-Type, 5/8-Inch Bolts: Bureau of Mines Rept. of Investigations 5228, 1956, 13 pp.

37/ Thomas, Edward, Progress and Problems in Roof Support: Coal Age, vol. 61, No. 8, August 1956, pp. 85-87.

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The equation can be used when designing roof-bolting systems and provides a means of determining how much support is furnished by various bolting plans rather than indicating how much support is needed. If an existing bolting system is inadequate, the formula can be used to determine the present reinforcement factor and the most effective way to increase it. Application of the equations is simplified when the physical properties of the rock are determined beforehand.

Under a cooperative agreement with the Youngstown Mines Corp., the facilities of the Dehue mine (W. Va.) were made available to the Bureau for studies relating to all phases of roof-bolting.

Acid Mine Waters

Huge volumes of mine water from underground workings are pumped, drained, and stored. Data were obtained on the volume and quality of this water and on the corrosion- and erosion-resistant properties of 25 different metals and alloys exposed to attack by the most highly acid mine water discharged in the anthracite region. The most highly corrosive mine waters available for tests were those from the Loree mine of The Hudson Coal Co., Plymouth, Pa., in the Northern Field.38/

On the basis of the immersion and revolving-spindle tests, types 302, 303, 304, 316, 410, 430, 446, Armco 17-4 PH, and Armco 17-7 PH stainless steels; cast stainless-steel alloys (ACI) CE30, CF8M, and HC; and titanium have adequate corrosion resistance. Specimens of these materials showed no corrosion, although some tests lasted 150 days. The corrosion rate of 89-2-9 bronze (89 percent copper, 2 lead, 9 tin) was one-fourth that of 75-15-10 bronze (75 copper, 15 lead, 10 tin) generally used in pumping acid mine water. After 30 days' testing, the 89-2-9 bronze lost more than 300 times as much weight as type HC stainless steel (the highest average weight loss of stainless steels being 0.1 gram) after 150 days. Increased lead content of bronze appears to increase the rate of corrosion. The aluminum-bronze alloys corroded as fast or faster than 75-15-10 bronze. Steel and 5-percent-chromium-steel were dissolved completely during 19 days of testing, indicating that 11 to 12 percent of chromium is necessary for adequate resistance.

Mine Atmospheres

The analysis of mine atmospheres plays an important part in promoting safety and in maintaining safe conditions in mining operations - particularly in coal mining, where the flammable gas, methane, is a constant hazard in some mines and may be encountered unexpectedly in others.39 40/ Methane also

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38/ Ash, S. H., Dierks, H. A., Felegy, E. W., Huston, K. M., Kennedy, D. O., Miller, P. S., and Rosella, J. J., Corrosive and Erosive Effects of Acid Mine Waters on Metals and Alloys for Mine Pumping Equipment and Drainage Facilities: Bureau of Mines Bull. 555, 1955, 46 pp.

39/ Watson, H. A., and Berger, L. B., Equipment for Analyzing Mine Atmospheres, With Special Reference to Haldane-Type Apparatus, Bureau of Mines Inf. Circ. 7728, 1956, 51 pp.

40/ Stahl, R. W., and Dodge, C. H., Methane Buildup During Cutting and Continuous Mining Operations: Bureau of Mines Rept. of Investigations 5288, 1956, 22 pp.

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has been found, often with disastrous results, in noncoal mines and in tunnels under construction. Other conditions that must be considered in respect to underground atmospheres are accumulations of carbon dioxide, depletion of oxygen, and presence of toxic gases such as carbon monoxide and oxides of nitrogen. The Haldane apparatus is well suited to routine determination of gaseous constituents in normal mine atmospheres - considering limited volume of sample generally available, time required for analysis, and attainable accuracy of results. The portable Orsat apparatus is suitable for determining carbon dioxide, oxygen, carbon monoxide, methane, and nitrogen in the atmospheres of sealed areas or for analyzing oxygen-deficient atmospheres or considerable accumulations of carbon dioxide, methane, or nitrogen in unventilated places. The laboratory-type Orsat apparatus has the same field of application but is more versatile and accurate.

The pyrotannic acid method is suitable for determining low concentrations (0.01 to 0.20 percent) of carbon monoxide in the air of ventilated places or in the gases from sealed fire areas when the fire is nearly extinguished and the area is cooling. The phosphomolybdic acid method is more sensitive (0.002 to 0.06 percent) and accurate, but the apparatus and the technique also are more complicated.

The phenoldisulfonic acid method is suitable for determining oxides of nitrogen in concentrations that may be encountered (1) in the air of working places after blasting, (2) from the use of internal-combustion (diesel) engines, or (3) in comparatively high concentrations such as may be present in the undiluted exhaust gases of internal-combustion engines.

Certain components of gaseous mixtures may be determined by measuring their ability to absorb infrared radiation. Compounds such as carbon dioxide, carbon monoxide, water vapor, methane, and other hydrocarbons absorb infrared radiation, whereas symmetrical diatomic gases such as oxygen, nitrogen, and hydrogen do not. Different compounds exhibit characteristic absorption behavior when subjected to infrared radiation.

Rock Dusting

Mechanized mining has increased greatly the production and dissemination of fine coal dust in mine entries. The use of high-speed machines has increased the probability of incendive sparks, which are produced by frictional contact between moving metals and certain mineral substances in the coal seam or in adjoining rock strata. In gassy seams rapid extraction of coal has increased the rate of gas evolution.

These conditions have made more urgent the need of preventing the ignition of coal dust. The propagation of coal-dust flames can be arrested and the development of widespread explosions prevented by properly applied, generalized rock dusting. To correct serious misconceptions regarding the action of rock dust and the development of mine explosions, a Bureau of Mines Information Circular[41/] has been published giving detailed explanations of

41/ Hartmann, Irving, and Westfield, James, Rock Dusting and Sampling, Including Wet Rock Dusting, at the Bureau of Mines Experimental Coal Mine: Bureau of Mines Inf. Circ. 7755, 1956, 13 pp.

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hazardous dust conditions and how they may be remedied. Recommendations are stated for applying rock dust effectively by both dry and wet methods, and procedures for sampling the rock-dusted areas are discussed.

Dust Control in Mines

Information available from Bureau of Mines investigations and from published and unpublished reports has been reviewed and summarized to outline current practice in dust control in and around mines, tunnels, and quarries.42/

Preparation and crushing plants have made preliminary tests of fine materials to determine the best type of dust-control equipment, quantities of air, number of sprays, kind of dust-allaying fluids, and location of hoods or enclosures. Advance planning has paid for itself in more effective control. Similar planning in a tunnel resulted in healthful conditions and low concentrations of dust.

Underground work on dust control in mines and tunnels has been centered on better wet drilling and pneumatic collection of dust during drilling. Some of the newer methods of drilling the roof in coal mines have featured the use of a tube around the drill steel through which the dust is drawn, extracting drill cuttings through hollow drill steel, and combining rotary drills with a positive-displacement electrically driven pump and collecting hood. Water infusion has been adopted in only one mine in the United States. Attention also has been given to better ventilation as a means of dust control, especially where continuous-type mining machines are used. An exhaust fan and tubing were tried in one mine.

Probably the greatest interest in the dust problem has been in sampling, especially with the new molecular-membrane filter. Other materials, such as Fiberglas, have been investigated also. Less fatiguing methods have been developed for counting dust. A so-called absolute method consists of collecting dust on a molecular filter, photographing the dust in an electron microscope, then measuring the particles as projected on a ruled screen. Another method, called the most-probable-number method, reportedly has an accuracy close to that of conventional counting. Improved sampling instruments include a rotating and oscillating thermal precipitator and a portable electrostatic precipitator that collects samples on glass or metal slides, has a smaller lightweight head, and operates from 100 volts a.c. or 6 volts d.c. A five-stage cascade impactor has been developed in which properly sized openings give high collection efficiency.

Details of a survey (1954) in 169 anthracite mines and in 3,413 bituminous coal and lignite mines are given in a Bureau of Mines Information Circular43/ that presents data on the extent to which dust control is practiced in the coal-mining industry.

42/ Owings, C. W., Dust Control in Mining, Tunneling, and Quarrying in the United States: Bureau of Mines Inf. Circ. 7760, 1956, 38 pp.

43/ Barnes, R. W., Gregory, M. J., Owings, C. W., and Berger, L. B., Survey of Dust-Control Practices in the Coal-Mining Industry: Bureau of Mines Inf. Circ. 7733, 1956, 49 pp.

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One phase of Bureau of Mines health and safety work is promoting the use of safe and effective dust-collecting devices while rock drilling in coal mines. To insure that adequate equipment of this type is available to the coal-mining industry, the Bureau of Mines conducts investigations and issues approvals for dust-collecting equipment that meets the requirements of a schedule established for testing dust collectors intended for use while drilling the roof for bolting and during other rock drilling. Performance tests are conducted under the provisions of this schedule by engineers of the Branch of Health Research.

An Information Circular describes briefly the requirements and procedure for approval and lists drill-dust collectors that have been approved.44/

Electrical Equipment

The current investigations of electrical equipment reflect a strong trend toward concentrated systems in coal mining with continuous miners, bridge conveyors, extensible conveyor belts, and cable-reel shuttle cars displacing conventional cutting machines, loading machines, and drilling machines. On April 26, 1949, the Bureau first approved a machine designed to replace the cycle of cuttings that blasted and loaded coal in a single operation, then delivered it to transportation equipment directly from the solid face. By January 1, 1955, the Bureau had granted 30 approvals for designs of machines of this type. An Information Circular describing the approved machines has been published.45/ Where older systems of mining prevail, the Bureau is interested in promoting multiple blasting with short-delay (millisecond) detonators because of safety and other advantages. The various factors involved have been studied to where revision of the present schedule for the approval of multiple-shot blasting machines is in order.

The possibility of generalized mine lighting in gassy coal mines became a reality in 1956. A schedule previously developed to permit approval of lighting systems became official on January 3, 1956; and on October 30, two systems - one for connection to grounded distribution systems and one for connection to ungrounded isolated circuits - were approved. The lamp fixtures for either system contain two 14-watt fluorescent tubes. The Bureau also was responsible for designing an explosionproof enclosure for a 1,000-watt color-corrected lamp suitable for underground motion-picture photography in gassy mines. Another design was an explosionproof enclosure for flashbulbs - also for underground photography. Installation of the new lighting systems is expected to result in greater safety and production.

Late in 1955, Schedule 28, covering test and acceptance of fire-resistant conveyor belts, was issued. Since then, on the basis of examination and test, more than 150 conveyor belts, the products of 15 manufacturers, have

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44/ Owings, C. W., Anderson, F. G., Harmon, J. P., Johnson, L., and Berger, L. B., Drill-Dust Collectors Approved by the Bureau of Mines as of January 31, 1956: Bureau of Mines Inf. Circ. 7741, 1956, 25 pp.

45/ Brunot, H. V., Permissible Mining-Loading Equipment: Bureau of Mines Inf. Circ. 7736, 1956, 33 pp.

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been accepted and listed as fire-resistant. A report describing recommended safety features and controls of belt-installations has been published as an Information Circular to assist manufacturers and mine operators in meeting improved standards.46/

A total of 96 units of different types of electrical equipment was approved for coal-mining service during 1956.

Health Research

Health research was directed toward the study and evaluation of conditions affecting the working environment in coal mines and other mineral industries and toward the promotion of safe and healthful conditions. Information was published on the occurrence and control of harmful gases and dusts, on tests of equipment for protection against such atmospheric contaminants, and on the study and improvement of mine ventilation and other factors that affect the quality of the air in underground workings.

A portable interferometer-type methane indicator was studied for use in coal mines. The instrument proved accurate for detecting methane in otherwise normal air, but when the proportions of oxygen and nitrogen in the sampled atmosphere differed significantly from those of normal air, methane was indicated in error. This behavior is considered typical of instruments of the interferometer type. Interfering gases such as carbon dioxide and water vapor were removed satisfactorily by suitable scrubbing media.

In developing requirements for fire-resistant conveyor belts for use in coal mines, comparative experiments were conducted to determine the nature and quantity of gases produced by burning or thermal decomposition of rubber, Neoprene, and polyvinylchloride. Preliminary results of this work were presented at the Ninth International Conference of Directors of Safety in Mines Research, Heerlen, Netherlands, and Brussels, Belgium, June 28 to July 4, 1956.

As part of a study to explore the possibility of substituting nonflammable fluids for the flammable liquids that generally are used in the hydraulic systems of coal-mining machinery, viscosity determinations and empirical flammability tests were conducted on various materials such as oil-water emulsions, water-soluble oils, and other modified materials that might possess desirable qualities from the standpoint of nonflammability and yet not be unreasonably expensive.

An experimental air cleaner, used in conjunction with a continuous-type coal-mining machine, was studied in the field. Although this device was in the development stage and not completely effective, it is considered a step in the right direction, as it represents an effort on the part of a mining-machine manufacturer to "build in" dust control as part of his equipment. The control of dust produced by continuous-type coal-mining machines was viewed by the American Mining Congress as important enough to warrant establishment of a subcommittee, which included personnel of the Branch of Health Research of the Bureau, to study the problem.

46/ Brown, C. L., Safety Aspects of Controls and Operations of Belt Conveyors in Coal Mines: Bureau of Mines Inf. Circ. 7749, 1956, 15 pp.

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Development work was conducted on apparatus intended to be intrinsically safe for sampling airborne dust in gassy mines. This work included completion of a motor-driven pump for the impinger apparatus and an oscillating thermal precipitator and development of a high-volume sampler to where it shows promise of satisfactory performance. The high-volume sampler is needed particularly to obtain samples of airborne dust in the amounts required for X-ray diffraction.

Testing respiratory protective equipment, including gas masks and dust respirators applicable to use in coal mines, resulted in 1 new approval and 33 extensions of approval covering modifications and improvements of design during the year. As part of the Bureau program of coal-mine inspection under the provisions of the Federal Coal Mine Safety Act, approximately 19,000 samples of mine air were analyzed to determine the adequacy of ventilation in coal mines, detect and aid in eliminating the hazards from flammable and toxic gases in mines, provide the information necessary for controlling and extinguishing fires in coal mines, and for other investigations in coal-mining operations.

The Bureau participated in revision of an American Standard safety code that will provide valuable guidance to those concerned with respiratory protective equipment.

Ventilation surveys were conducted by absolute pressure methods, which reveal zones of above-average resistance to airflow in the overall ventilation pattern of the mine and therefore provide information applicable to the improvement of ventilating efficiency. An electric analog developed for solving mine-ventilation-network problems has proved valuable when using the results of the quantity and pressure surveys in predicting the results of proposed changes in methods of coal-mine ventilation. In several instances such analyses and predictions resulted in savings to the mining companies concerned. A study was completed of the effectiveness of bleeder entries in ventilating pillared areas in coal mines to prevent or reduce dangerous accumulations of methane in such areas.

Roof-Fall Fatalities

The 355 deaths in 1955 due to accidents at bituminous coal mines were investigated; of the 299 that occurred underground, falls of roof were responsible for 194, or 65 percent.47/ In 1954 falls of roof, face, or rib caused 177 deaths or 60 percent of all underground fatalities in bituminous coal mines. The rate of occurrence in 1955, as in 1954, was 0.53 fatalities per million man-hours.

Ninety-five percent of the mines in which roof-fall fatalities occurred had timbering plans, but in 48 percent of these the timbering plan was not being followed. Fifty-six percent of the fatalities was inby the permanent supports, and 58 percent in places equipped for mechanical loading. Sixteen percent occurred where roof bolting was specified as the roof-support medium.

47/ Maize, E. R., and Wallace, J. J., Falls of Roof, The No. 1 Killer at Bituminous-Coal Mines: Bureau of Mines HSS 445, Mineral Industry Surveys 1955, 9 pp.

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Fourteen fatal accidents were reported in 1955 where roof bolting was the accepted method of roof support, and all were within the 25-foot face zone where approximately three-fourths of all roof-fall injuries occur. In 9 of the 14 fatalities attributed to roof-bolted areas the rock that fell was in the unsupported space between the last row of bolts and the face being mined.

Sixty-six hand loaders, including six owners and two foremen performing loading operations, were killed by roof falls. The latter 8 were killed in 8 mines employing a total of 30 men, each 1 to 5 employees. This group had 23 fatalities, or 12 percent of the total for the year.

Reports of 60 underground fatalities at the anthracite mines of Pennsylvania in 1955[48/] showed that roof-fall accidents caused 34 deaths, or 57 percent of the total. Haulage accidents caused eight deaths; a breaker collapsed and caused four deaths; a gas explosion caused two; explosives, two; falls of person, two; electricity, one; and miscellaneous, four. Three others were at stripping operations.

In seven working places where face, rib, or foof-fall fatalities occurred an official was the victim or was nearby when the accident happened. In six other working areas where roof-fall fatalities occurred an official had examined the working place less than 30 minutes before the fatality; therefore, an official was in or had just left the working area in 38 percent of the cases where face, rib, or roof-fall accidents occurred. These data indicate the need for more adequate examinations by officials if such accidents are to be curtailed.

Although roof testing is important, judgment should not be substituted for the minimum supports required to comply with a systematic roof-support plan; rather, testing the roof should serve to determine whether additional roof supports in excess of the regular timbering plan are necessary.

Analysis of Coal-Mine-Accident Data

A statistical analysis and report of information from coal-producing companies and State mine officials on deaths and injuries to persons in the coal-mining industry for 1952 showed more favorable experience than in 1951, when the best previous record was reported.[49/]

Safety Training and Education

Health and Safety activities of the Bureau of Mines in coal mining combine functions of research, investigation, education, and inspection.[50/]

48/ Mather, J. V., Fatalities at Pennsylvania Anthracite Mines, 1955 Bureau of Mines: Bureau of Mines HSS 444, Mineral Industry Surveys, 1956, 25 pp.

49/ Reese, S. T., Wrenn, V. E., and Reid, E. J., Injury Experience in Coal Mining, 1952: Bureau of Mines Bull. 559, 1955 60 pp.

50/ Westfield, James, Health and Safety Activities of the Bureau of Mines, Fiscal Year 1955: Bureau of Mines Inf. Circ. 7732, 1956, 21 pp.

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Often the performance of service or inspection duties is interwoven with the investigation of hazards and accidents or research in combating mine fires and explosions.51 52/

Representatives of the Bureau participated in meetings on safety in coal mining and contributed to an international conference of leaders in coal-mine-safety research.53/ Bureau participation in national meetings is exemplified by the work done at the National First Aid and Mine Rescue Contest at Knoxville, Tenn.54/

A safety educational course for coal-mine supervisors and employees has been conducted for more than 10 years. Revisions of the text are necessary every few years because of new techniques, new equipment, and better information gained by continuing investigations. One section of this course, pertaining to electrical accidents, was revised in 1956.55/ Information on health and safety publications of the Bureau of Mines was published as a bulletin.56/

STORAGE AND PREPARATION

Storage of Lignite

Because lignite tends to weather and slack, domestic sizes are never stored for long periods. Industrial consumers maintain emergency stockpiles, although storage creates a problem because of spontaneous heating.

A method of safe storage has been developed in cooperation with the U.S. Army Corps of Engineers at Garrison Dam, Riverdale, N. Dak. Over 2 million tons of lignite is stored in eight piles. Periodic observation during the past year continues to reflect stable, safe storage conditions inside the stockpiles. Seasonal temperatures and infiltration of air at the surface of the piles effect changes at a slow, safe rate. Yearly determinations of heating values showed a decrease of less than 1 percent from August 1953 to September 1956. The major difficulty of storage continues to be erosion by wind and rain. No reoccurrence of heating in accumulated slack drifts was observed during the past year. Snow fences installed in three parallel rows

51/ Westfield, James, Weaver, H. F., and Keenan, C. M., Administration of the Federal Coal-Mine Safety Act, Calendar Year 1954: Bureau of Mines Inf. Circ. 7734, 1956, 73 pp.

52/ Westfield, James, Weaver, H. F., and Keenan, C. M., Administration of the Federal Coal-Mine Safety Act, Calendar Year 1955: Bureau of Mines Inf. Circ. 7765, 1956, 71 pp.

53/ Gleim, E. J., International Cooperation in Reducing Mine Hazards: Bureau of Mines Inf. Circ. 7739, 1956, 15 pp.

54/ Tomlinson, W. H., National First-Aid and Mine Rescue Contest, Knoxville, Tenn., October 10, 11, and 12, 1955: Bureau of Mines Inf. Circ. 7764. 1956, 83 pp.

55/ Electrical Accidents in Bituminous-Coal Mines: Bureau of Mines Miners' Circ. 59 (1954 revision), 1955, 100 pp.

56/ Humphrey, H. B., and Stratton, H. J., Bibliography of Bureau of Mines Health and Safety Publications, January 1947-June 1955: Bureau of Mines Bull. 558, 1956, 89 pp.

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on one pile have been partly effective in controlling the deposition of slack. The accumulation of drifts on the sides of the pile has been reduced, and slack can be spread readily at the fences. More significant, however, is the growth of weed on the sides and tops of the piles. This growth aids in controlling wind erosion and should be encouraged as an effective and inexpensive method of reducing maintenance problems.

Freezeproofing of Lignite

Despite severe winter weather in the North Central region, lignite is mined and transported as the consumer requires it. Industrial fuel frequently agglomerates by freezing in transport, which results in increased handling and unloading costs. Oil treatment at the mine somewhat reduces unloading difficulties.

During the past year a method was developed for measuring the degree of agglomeration by freezing laboratory samples. Two methods were unsatisfactory: (1) Measuring the impact on the container wall required to dislodge an agglomerated sample and (2) determining the compression required to fracture a cylindrical agglomerated sample. In a third method providing satisfactory reproducibility samples are ruptured by inflating imbedded pieces of rubber tubing. The sample containers are cylindrical, 1 foot high and 1 square foot in cross section. Both the top and the wall are removable. A copper tube extends through and perpendicular to the center of the bottom. Rubber tubing is threaded over the copper tube and clamped so that a 4-inch length is free to expand at the center of the sample. Following agglomeration, the top and the wall of the container are removed. Compressed nitrogen, used to expand the tubing, is admitted through holes drilled in the copper tube. Pressure is increased at a controlled rate until the sample is ruptured, and the maximum pressure is used as a measure of agglomeration. A ruptured sample and the expanded tubing are shown in figure 8. A total of 20 containers can be placed in the refrigerated chamber in any single test.

Satisfactory reproducibility has been achieved by closely controlling the sample and procedure. Factors influencing agglomeration by freezing and the various freezeproofing agents are being evaluated.

Moisture in Lignite

The results of a study of this subject were published in April 1956 and were reviewed in the previous annual report on technologic work on coal.57/

Size Reduction of Lignite

Pulverizing lignite presents much greater problems than reducing it to coarse sizes. Emphasis in the study of size reduction continues therefore on pulverizing characteristics and power requirements.

57/ Hoeppner, J. J., Fowkes, W. W., and McMurtrie, R., Removal of Moisture From Lignite in Inert-Gas Atmospheres: Bureau of Mines Rept. of Investigations 5215, 1956, 25 pp.

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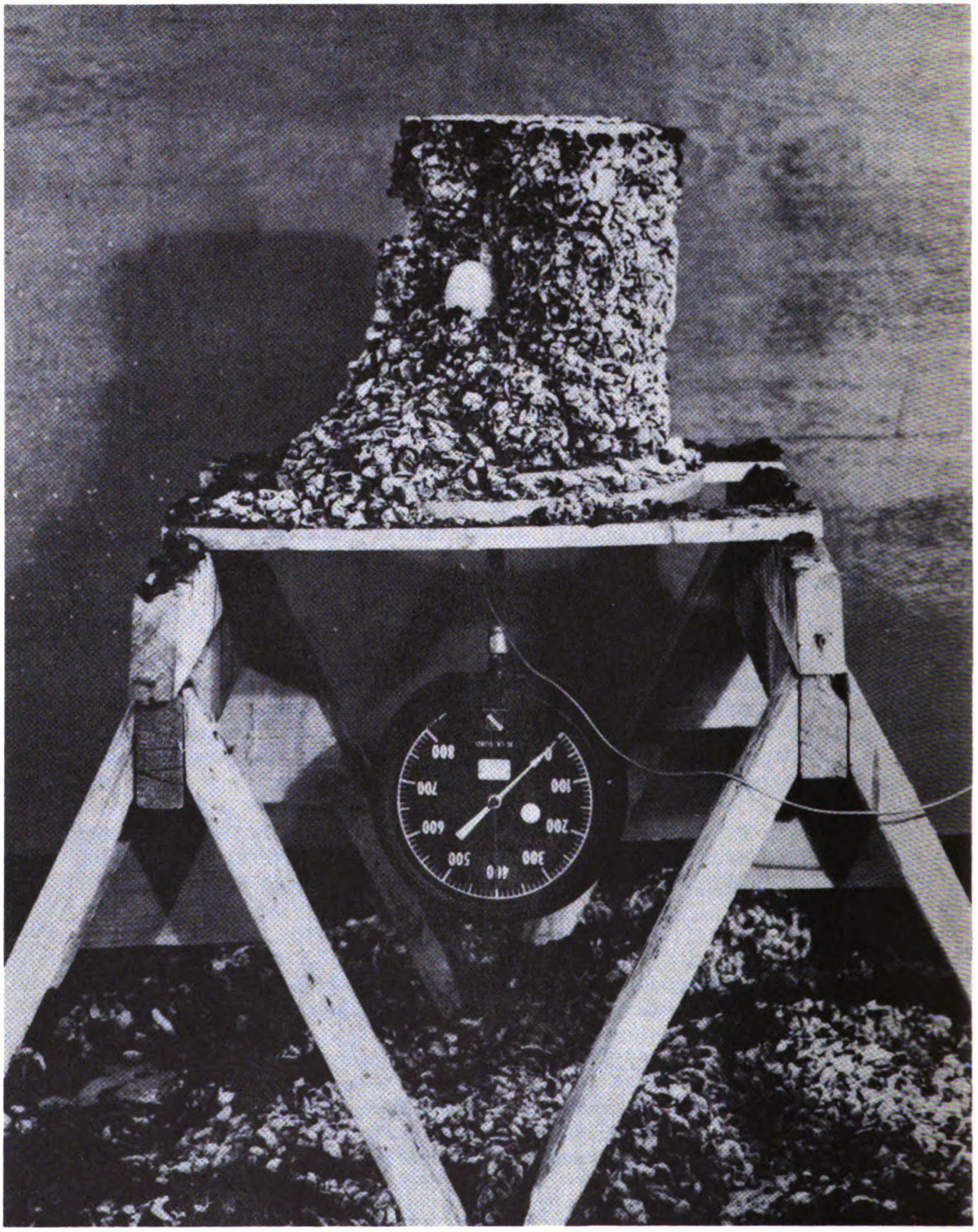

FIGURE 8. - Frozen Lignite Ruptured by Expansion of Rubber Tubing.

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During the past year, earlier field tests were supplemented by laboratory studies of an experimental air-swept impact pulverizer, loaned by the University of North Dakota. Despite close control of operating variables, stable operation has not yet been achieved. Material builds up progressively in the pulverizer and prevents reproducibility of operation. A possible cause of the buildup is the high moisture content of lignite. Efforts are being made to eliminate unstable operation without the use of heated air. In many applications pulverized firing methods could be adopted if air heaters can be eliminated.

Mechanism of Flotation

The most difficult steps in coal preparation are cleaning and dewatering the very fine sizes. Since this material cannot be treated satisfactorily by processes that depend on differences in specific gravities of coal and associated impurities, some other property must be utilized. Present indications are that processes depending on differences in wetting characteristics are most suitable for commercial exploitation. The Bureau of Mines is therefore studying the theory and practice of flotation of coal.

During the year, particular emphasis has been placed on explaining the mechanism of froth and kerosine flotation and determining the influence of operating variables on cell capacity. These problems are closely related, as rational selection of cell design and operating conditions for maximum capacity must be based upon a mechanism of flotation.

Recent work showed that, contrary to experience with flotation of minerals, supercharging markedly decreased cell capacity when fine coal was floated with kerosine as the principal reagent. Also, that baffle blocks around the periphery of the impeller increased cell capacity, although power consumption was simultaneously decreased. One therefore may assume that the mechanism of kerosine flotation of coal is somewhat different from that of ordinary froth flotation of coal. Such an assumption is consistent with the striking difference in appearance and stability of the float products from these processes. Thus, whereas flotation of coal with a frothing agent only yielded a product that readily overflowed from the cell and broke down almost instantly, kerosine flotation yielded a dense, matlike product that had to be removed from the cell with drags or scrapers and that remained stable for an indefinite period.

When tests were conducted in which the air intakes in the standpipe were sealed and the only air entering the cell was that accompanying the pulp and leaking into the standpipe, significant increases in cell capacity resulted, quite similar to those obtained with baffle blocks. An instrument devised especially to permit measurement of the rate of gas evolution from the surface of the cell indicated that these blocks markedly reduced aeration of the pulp and therefore may serve to increase cell capacities by a similar mechanism. Further experiments supported the tentative conclusion that, depending on the particular coal and operating conditions, almost any change (within limits) that causes a substantial reduction in the rate of aeration is accompanied by an increase in cell capacity. Because of the success of

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supercharging in a wide range of mineral-flotation processes further work is needed to delineate completely the differences in these processes.

Laboratory studies of factors influencing flotation rates have been initiated, and preliminary determinations of the effects of variations in temperature, particle size, surface oxidation, contact angle, and pulp density have been completed. Results of tests on one coal show that certain alcohols, such as methyl isobutyl carbinol, normal amyl alcohol, and tertiary amyl alcohol, are highly sensitive to changes in temperature - an increase of 30° C. causes approximately a fourfold decrease in the first-order flotation-rate constants. Other reagents, including metacresol, kerosine, ethylene glycol, and diethylene glycol, were relatively insensitive to changes in temperature.

Fine-Coal Cleaning

Cleaning fine coal presents more difficult problems, both technical and economic, than treating the coarser sizes; moreover, these problems are intensified by the greater proportion of fine coal that results from today's mining methods. Thus much Bureau research in coal cleaning is directed toward improved means of cleaning fine coal.

Investigation of the feldspar jig, used almost universally in Europe, has progressed from laboratory to full-scale operation. The first modern feldspar jig to be used in this country was installed recently at a mine in the State of Washington. Studying its operation presents an excellent opportunity to evaluate its applicability to American coals. The jig, an 80-ton-per-hour Baum-type unit, of American manufacture, is treating 1/4-inch coal at a rate greater than 1 ton per hour per square foot of bed area. Performance data are being obtained with one coal that is unusually difficult to clean and another comparatively easy to clean.

Another investigation is directed toward utilizing the dense-medium process for treating fine coal. Although fine coal and its associated impurities can be sharply separated with dense medium in a cyclone, problems of recovering coal from the medium, conditioning the medium for reuse, and minimizing consumption of the medium remain to be solved before commercial application.

Recovery of Solids From Washery Water

Recovering fine solids from washery water assumes steadily increasing importance as stream-pollution control becomes more stringent. The Bureau is continuing its study of the efficiency of liquid-solids separation by various types of equipment that may be used to free washery water from contaminating slimes. During the past year, flocculants and vacuum filtration were investigated. Some slurries, virtually unfilterable before treatment because of their clay content, were easily filtered after flocculation.

Coarse-Coal Cleaning

A cone-type dense-medium pilot plant with a nominal capacity of 10 tons per hour has been used in a number of tests of Pittsburgh-bed coal. Six of

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these tests have been described in detail.[58/] In two tests grade B magnetite was the medium; in four tests, pulverized refuse was used. The refuse was sink material from a commercial dense-medium separator and consisted of clean shale and laminated coal and shale. Magnetite was the superior medium, but pulverized refuse, which might be available locally at low cost, can be used under some conditions. When sizes coarser than 1 inch were cleaned at low specific gravities of separation, pulverized refuse compared favorably with magnetite. Such a medium could be recovered by froth-flotation cells and thickeners, or a portion could be discarded continuously and new medium added when refuse is available at low cost.

Viscosities of suspensions containing pulverized refuse and water increased with specific gravity; they became so high and the rate of increase so rapid at a specific gravity of about 1.45 that separation at higher specific gravities would be difficult.

Low-Ash Coal for Electrode Carbon

An investigation has been completed of means for preparing superclean coal that could be used as a source of electrode carbon for producing aluminum.[59/] The rapidly growing aluminum industry demands an ever-increasing supply of carbon for electrodes; consumption is about 1,300 pounds for each ton of metal produced. Current United States requirements are met by petroleum coke, but changing markets and shifts in petroleum-refining methods suggest that future needs may have to be met by coal prepared to exacting standards of purity.

Fifteen coals from Alabama, Kentucky, Utah, Washington, West Virginia, and Wyoming were subjected to three techniques for removing impurities: (1) Physical methods such as float-and-sink separation and froth flotation, (2) leaching with various acids and alkalies, and (3) solvation of coal in heavy-residue creosote.

Physical cleaning methods did not yield material of satisfactory purity from any of the coals tested; only one coal could be cleaned to less than 1.0 percent ash; however, physical cleaning proved advantageous as a pretreatment for chemical processing. Leaching with hydrochloric and hydrofluoric acids gave products containing as little as 0.35 percent ash (when calculated to a coke basis). Solvation was even more successful and yielded coal that would provide coke containing as little as 0.15 percent ash. The analyses of two treated coals and that of petroleum coke are as follows:

58/ Palowitch, E. R., Sokaski, Michael, and Jolley, T. R., Dense-Medium Pilot-Plant Performance Tests on Coal: Bureau of Mines Rept. of Investigations 5212, 1956, 52 pp.

59/ Campbell, R. J., Jr., Hilton, R. J., and Boyd, C. L., Coal as a Source of Electrode Carbon in Aluminum Production: Bureau of Mines Rept. of Investigations 5191, 1956, 53 pp.

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Comparison of West Virginia coals prepared by solvation (calculated to coke basis) with calcined petroleum coke

	Carswell (Pocahontas No. 3 coal)	Margarette (Sewell coal)	Calcined petroleum coke
Total ash.....percent	0.15	0.18	0.34
Iron.....do...	.036	.022	.050
Silicon.....do...	.016	.032	.029
Calcium.....do...	.021	.016	.042

Techniques employed in solvation cleaning are established in other industries, and standard equipment probably could be used with only minor modification in commercial application. Costs of energy, equipment, and solvent are thought to be low; however, the economics of the process would have to be estimated from pilot-plant operation.

Preparation Characteristics of Coking Coal

A survey of the preparation characteristics of coking coal in the United States was begun in 1948 as the second phase of a Bureau investigation to evaluate reserves of coking coal. Its purpose was to study methods of upgrading marginal coal through effective preparation. The study is being made to determine:

1. Which coals, either as mined or after beneficiation by conventional preparation, are suited chemically under present standards for producing metallurgical coke.

2. Which coals require special or more intensive preparation than now is being practiced.

3. Which coals cannot be considered potential sources of metallurgical coke regardless of extent of preparation.

According to present standards for coal for making metallurgical coke, it is believed generally that the sulfur content on a dry basis should not exceed 1.25 percent. Coal with more than this amount can and has been used for making metallurgical coke, especially when blended with coal containing less sulfur. In this series of reports 1.25 percent of sulfur is used as the upper limit.

Coals are being studied for preparation characteristics on a county basis, initially in the Appalachian area. During 1956, reports were published for two counties, one in Kentucky and one in West Virginia. Figure 9 indicates the 18 counties for which Reports of Investigations now are available, the numbers of the reports, and the status of unfinished reports.

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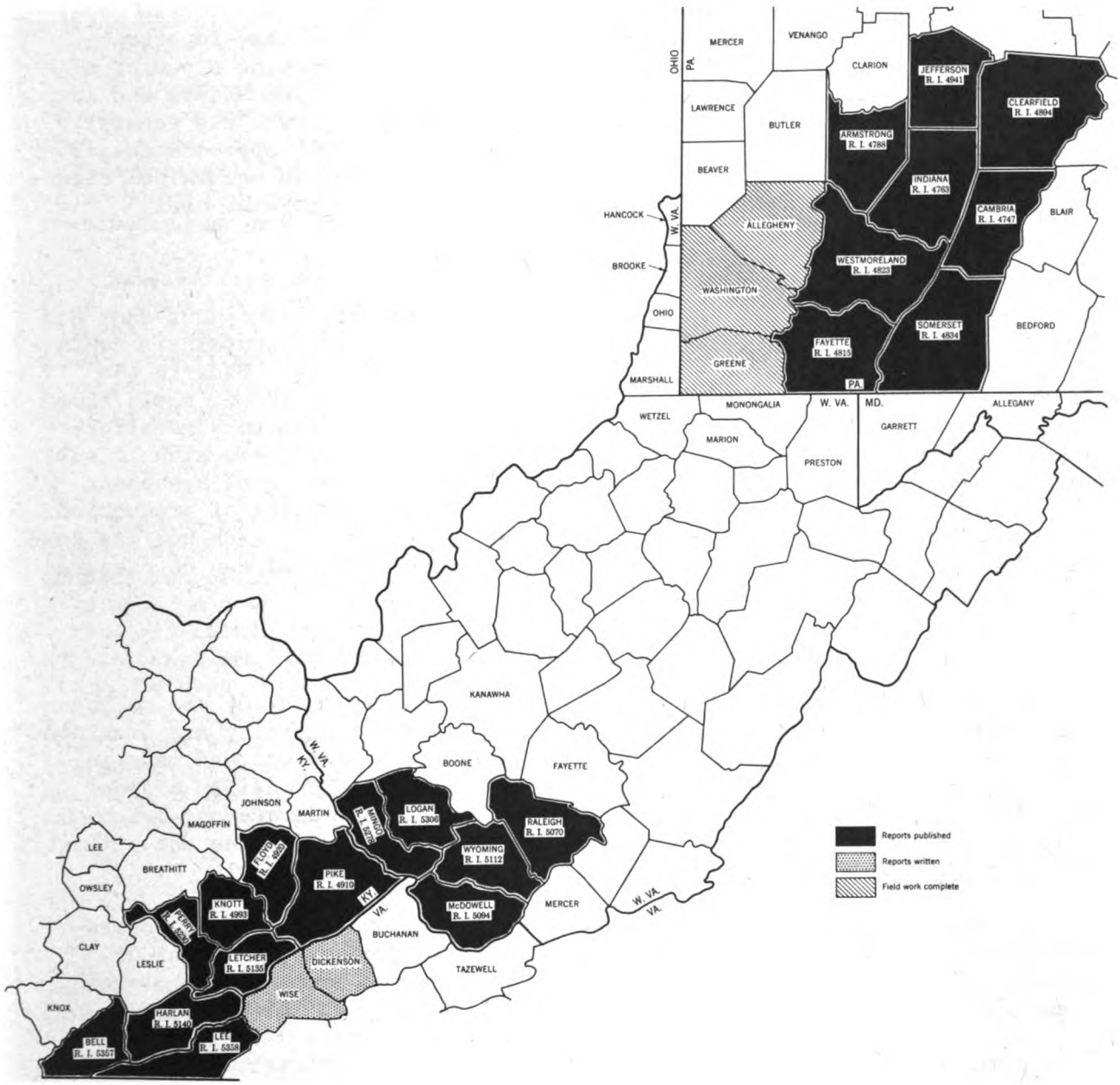

FIGURE 9. - Map Showing Status of Preparation Reports on Coking Coal.

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Perry County, Ky.

The Fire Clay and Flag beds are the principal sources of coal production in Perry County, Ky.60/ Both coals can be easily prepared to produce excellent metallurgical coal. The Hindman and Haddix beds are less important sources. The Hindman bed shows little possibility of becoming a source of metallurgical coal because it contains too much sulfur. The Haddix bed is well suited chemically for metallurgical use. Perry County coals generally are not responsive to upgrading by fine crushing. In 1953 approximately 30 percent of the coal produced in Perry County was cleaned in mechanical cleaning plants. The Flag bed was the source of most of the cleaned coal.

Mingo County, W. Va.

Coals of Mingo County, W. Va., are of high-volatile bituminous rank and generally have excellent coking properties.61/ The county contains a coking-coal reserve of great value to the iron and steel industry. Beds in the Cedar Grove group are the major source of production. Of these, coals from all but Upper Cedar Grove bed will, when washed, be satisfactory metallurgical-grade fuels. The Upper Cedar Grove coal was slightly too high in sulfur to meet the standard of 1.25 percent maximum, but this coal has been blended and used to make coke of good quality. Minor sources of production are the Alma, Coalburg, No. 2 Gas, and Buffalo Creek beds. Each bed was sampled one or more times, and the data indicate that each coal can be prepared for chemically suitable metallurgical-grade fuel.

Preparation Characteristics of Fuel Coal

Although most Bureau washability examinations are made for the coking-coal survey of the Appalachian Region, other coals occasionally are examined when the results are of public interest. Such an examination was made in 1956 in Alaska, where preparation practice determines the quality of coal supplied for military use and influences conservation of Government-owned coal reserves. The coal was difficult to treat and required the use of highly efficient preparation equipment to prevent loss of salable coal.

Another investigation was made of coal from a mine in Utah on Government-owned coal lands. Here washing would permit mining large areas of impure coal that otherwise would be lost; therefore a heavy-medium cleaning plant was installed late in the year.

A third washability examination was made of a coal from the Horseshoe Basin field of Teton County, Idaho, about which comparatively little had been known. This deposit of bituminous coal is remote from any other field and thus is of potential interest for local use. The coal is amenable to

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60/ Miller, J. W., and Jolley, T. R., Preparation Characteristics of Coal From Perry County, Ky.: Bureau of Mines Rept. of Investigations 5230, 1956, 28 pp.

61/ Miller, J. W., and Jolley, T. R., Preparation Characteristics of Coal From Mingo County, W. Va.: Bureau of Mines Rept. of Investigations 5278, 1956, 35 pp.

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beneficiation; it yields a product of 6-percent ash at 1.60 specific gravity. Although high-volatile B bituminous in rank, the coal has a free swelling index of 2-1/2.

Anthracite Preparation

Preparation studies were initiated to determine the washability characteristics of anthracites representing breaker production in the four anthracite fields. The object of these studies is to determine the type of preparation that will be of the greatest value to the industry. In addition, the data should indicate the yields of anthracite obtainable at specific ash contents when new or improved preparation techniques are applied.

Samples from two breakers were cleaned at specific gravities ranging from 1.4 to 2.0 at 0.1 increments. The solutions were prepared from zinc chloride, carbon tetrachloride, kerosine, and bromoform. Tests have been completed except for analyses of the pea through Buckwheat No. 5 sizes from one breaker. Analytical work on these samples will be completed, and the washability studies resumed when personnel are available.

Hazards in Coal-Drying Plants

Hazards in coal-drying plants were studied.62/ A questionnaire provided preliminary information on coal-drying facilities at more than 270 preparation plants; visits were made or conferences held with operators of 13 plants; and full-scale tests were made at 6 plants, supplemented by laboratory tests of dust and gas samples collected at these plants. Serious shortcomings were apparent in the location of equipment, accumulation of flammable dust, high oxygen content of drying gas, lack of vents, exits, fire-fighting equipment, and lack of interlocking controls. Ten specific suggestions are made in the report concerning safe operating practices in thermal coal-drying plants. In addition, the Code for the Prevention of Dust Explosions in Coal Pneumatic Cleaning Plants, issued by the National Fire Protection Association, is mentioned.

COMBUSTION, BOILER-WATER TREATMENT, AND POLLUTION

Heat Transfer in Furnaces

Because the major portion of coal mined is used to generate heat and power by combustion in furnaces, appreciable savings in the quantities used and in the capital costs associated with its use may result from a better understanding of the factors that influence the performance of a furnace. Activities in combustion of coal included measurement and correlation of the performance of various types of industrial and electric-utility boiler furnaces; development of a method of computing theoretical flame temperatures; and studies of the factors that cause ash deposits to build up on the external surfaces of boiler tubes, which results in serious reduction in boiler-heat absorption and impedes the flow of gases through the furnace.

62/ Brown, H. R., Dalzell, C. J., Hartmann, I., Toothman, G. J. R., and Schwartz, C. H., Fire and Explosion Hazards in Thermal Coal-Drying Plants: Bureau of Mines Rept. of Investigations 5198, 1956, 20 pp.

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Measurements of furnace performances of five large boilers were analyzed according to five previously proposed correlations. This analysis[63 64] showed that none of the published correlations is universally applicable to all types of furnaces and demonstrated the need for more accurate data. During the past year an additional industrial boiler has been tested in cooperation with the American Society of Mechanical Engineers (ASME). A continuing problem in this work is development of adequate instrumentation.[65]

A method of computing flame temperatures is of considerable value in designing a furnace scientifically and also may lead to more accurate correlations for predicting furnace performance. Although calculating a theoretical flame temperature requires only balancing the heating value of the fuel against the heat content of the product gases, estimating the latter term is complicated by the fact that at the high temperatures that prevail in flames several of the normally stable components, such as carbon dioxide and water, are partly dissociated into their elements with a concomitant loss of available heat. Computing this dissociation is an extremely lengthy operation, but a method has been developed that permits the use of the Remington Rand Univac-60 electronic computer, available at the Central Experiment Station. Calculations for 2,000 combinations of temperatures and excess air and fuel compositions have been processed into a few simple graphs and were presented before a meeting of the ASME.

A third aspect of furnace performance being studied is the cause and prevention of deposits on the fireside of boiler tubes. The mineral matter that occurs in coal may be released as fly ash, carried throughout the furnace, and deposited on one or more of the various heat-transfer surfaces. Several types of deposits may be formed, depending on the furnace and temperature and flow within the furnace. For each type of deposit, several mechanisms of formation have been suggested. These include theories that volatile components of the ash or of products of combustion are first condensed on tube surfaces and act as cementing agents for subsequent fly ash; that initial deposits of fly ash react with the flue gas to form the bond; and that certain components of the ash migrate in the solid phase and concentrate at the surface of the tube. This problem is being attacked on several fronts. An electric furnace capable of producing high-temperature gradients similar to those in an industrial unit has been constructed for studying the interactions of ash, flue gas, and high temperatures; chemical and X-ray analyses have been made of deposits secured from various functioning furnaces; and a field investigation has been conducted of an industrial furnace that was unusually free of deposits.

63/ Greyson, Murray, Mazie, G. P., Myers, J. W., Corey, R. C., and Graf, E. G., Evaluation of Factors Affecting Heat Transfer in Furnaces: Trans. ASME, vol. 78, No. 8, November 1956, pp. 1741-1746.

64/ Greyson, Murray, Mazie, G. P., Myers, J. W., Corey, R. C., and Graf, E. G., Evaluation of Factors Affecting Heat Transfer in Furnaces: Combustion, vol. 27, No. 8, February 1956, pp. 34-39.

65/ Corey, R. C., Measurement of Gas Temperatures With Thermocouples: Combustion, vol. 28, No. 4, October 1956, pp. 47-55.

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Fluid Mechanics

The performance of combustion equipment and almost all other devices in which chemical reaction and heat transfer take place is significantly affected by flow and momentum interchanges between fluid streams, solid reactants, and surfaces in the equipment. Fluid flow also is important in auxiliary operations of chemical and combustion systems, such as in pumping and transporting liquids, gases, and powder suspensions.

Limited time is being devoted to two aspects of this subject: An analysis of data obtained as part of other process studies, such as furnace performance, gasification, and incineration, to develop insight into the fluid dynamic behavior of systems and to obtain scaling-up factors; and a survey of technical literature, which has resulted in a review of developments during the previous year.66/

Fuel-Engineering Service

Advisory and field-testing services were continued to aid Federal agencies in selecting, testing, and using fuel and fuel-burning equipment, and in the economical use of steam. At the request of the various departments, as listed, several services were rendered. The Bureau assisted a military installation in Maryland during 1955 and 1956 in determining alterations needed in boiler and accessories to permit operation at full-rated load. At the Patuxent Naval Air Base, Potomac River Naval Command, advice was given on coal-burning difficulties at the base. Consideration also was given to the possibility of using these boilers at high rating for a proposed new hot-water heating system. Bureau assistance also was requested at Glenn Dale (Md.) Sanitarium in connection with a minor slagging problem in the boiler. At Cherry Point (N. C.) Marine Base, Bureau observations of power boilers operated on different size-consist coals led to recommendations of the most economical coal to be used during the next fiscal year. In preparation and during preliminary acceptance tests of a new 150,000-pound-per-hour power and heating boiler at the Naval Gun Factory, Potomac River Naval Command, the Bureau provided advisory services and personnel to supervise and assist in conducting performance and efficiency tests. The Bureau also analyzed coal, refuse, fly ash, etc., sampled during the tests and presently is rendering assistance to the Naval Gun Factory with a recommended program of correcting indicated deficiencies in measuring instruments and dust-collecting equipment.

Boiler-Water Treatment

Boiler-water research is designed to obtain new information and to apply both new and old information toward increasing fuel efficiency and decreasing fuel costs by improving operating procedures related to the use of water in Government-operated steam-generating plants. Suppressing deteriorating effects of the constituents of feed water, boiler water, and steam condensate minimizes corrosion, scale, and cracking in heating and power boilers and thus contributes toward decreased maintenance costs and increased safety.

66/ Weintraub, Murray, Flow of Fluids: Ind. Eng. Chem., vol. 48, No. 3, Pt. 2, March 1956, pp. 532-539.

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Condensate Corrosion

Area heating plants operated by Federal agencies frequently have several miles of condensate-return lines. In some instances serious corrosion has caused expensive losses of such valves and piping. Part of the expense stems from the loss of steam condensate, which not only represents a direct loss of fuel but also requires unusually large quantities of raw makeup water. Feedwater deaerators are not often sized to cope with large quantities of cold makeup, so that boilers are exposed to corrosive dissolved oxygen as well as to the potential scale- and sludge-forming mineral matter in raw water.

Treatment with neutralizing amines is a satisfactory method for controlling corrosion arising from high carbonic acid concentrations in the steam condensate, and several methods have been developed for effecting control with other chemicals such as the filming amines; however, these methods of treatment are expensive and hence are not used unless required.

A Bureau of Mines condensate-corrosion tester was developed to determine where preventive treatment is economically desirable. A report on the tester and its use for making surveys of return-line deterioration was presented at the 1956 annual meeting of the ASME.

The Bureau tester is principally a composite nipple comprising a set of six rings or shells that fit inside a specially machined pipe nipple. The average weight loss of the rings during exposure of the tester in the return-line system is evaluated in terms of an average corrosion index. The surface of the cleaned rings also shows the corrosion trend and thus indicates the factors responsible for corrosion. The tester therefore provides information on the corrosiveness of the steam condensate and also on the extent to which corrosion tends to be localized in the form of channels or pits. Figure 10 shows the component parts of the tester; the center nipple is 3 inches long

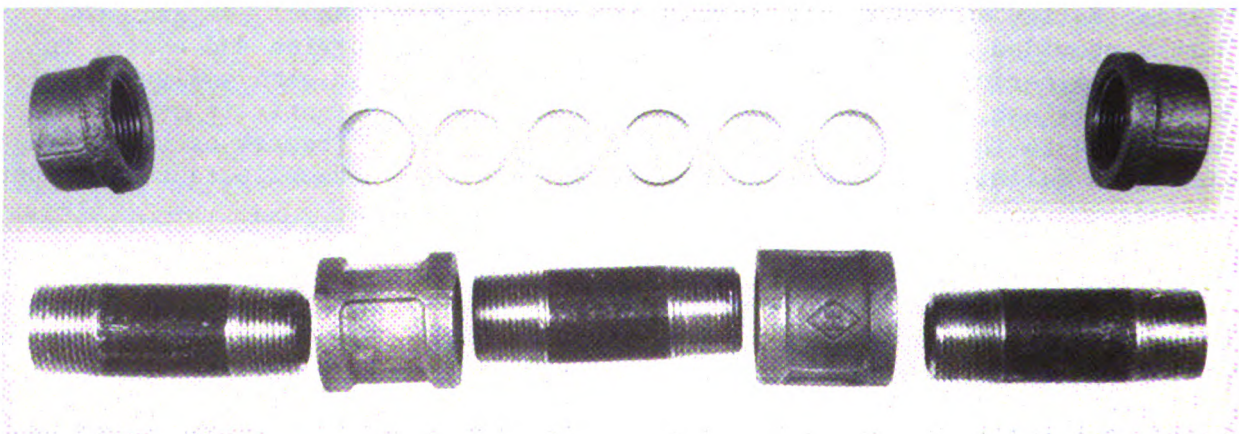

FIGURE 10. - Component Parts of Bureau of Mines Condensate-Corrosion Tester.

and receives 6-1/2-inch test rings of approximately Bessemer-steel composition. The tester is especially valuable for following improvements resulting

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from changes in operating practices and more especially for determining the extent to which chemical treatment has solved the corrosion problem.

During 1956 a total of 683 testers was distributed, and 328 returned testers evaluated. The field-test program has now been in effect long enough for the second phase - establishment of the effectiveness of chemical treatments - to be well underway.

When the survey discloses a corrosion problem, operating practices at the plant are reviewed as the first step toward solution. Control frequently is effected by decreasing the carbonate content of the feed water. When losses os steam and condensate are decreased, less mineral carbonate is imparted to the feed water by the makeup. In other instances external treatment has been used to remove part or substantially all carbonate from the raw water. Chemical treatment is prescribed only when it is justifiable economically. In one defense agency much of the corrosion problem was directly ascribable to carbon dioxide released in the boilers from the soda ash used for treating the boiler feed water. The agency had considered soda ash to be less hazardous to boilerroom personnel than sodium hydroxide. An agency directive ordered the use of sodium hydroxide instead of soda ash to control alkalinity. This change was effective and is expected to result in annual savings of about $1 million in maintenance costs.

Filming Treatment

Field surveys of corrosion showed that only partial success was being attained from octadecylamine-filming treatment for controlling corrosion in stream-condensate piping. Industrial plants apparently have experienced much more consistently satisfactory results. The greater length of the return-line system in Federal plants does not appear to be significant enough to explain the difference in effectiveness, especially as the results obtained at some large Government plants have been good. The material is known to steam-distill from the boiler and be transmitted by the condensate to the piping so as to effect filming and surface protection; however, little is known concerning factors that may retard distillation of steam or affect formation of film. The latter are especially important, since the chemical can be injected directly into the steam. Octadecylamine and octadecylamine acetate are available, but the relative advantages of the stability of each during recycling and the possible effects of the acetate group are unknown. As Federal plants would benefit from an effective filming treatment, a study was begun of the mobility and stability of the chemicals.

Before much could be learned about the movement of octadecylamine in the heating system, a method of analysis first was required. Second, a laboratory boiler was set up to study the volatility of the material in relation to the composition of boiler water. Third, a special corrosion train was designed to show the effects of condensate composition on the tendency of octadecylamine to form protective films. The final step will be applying this information to a study at a heating plant.

A method for determining octadecylamine in low concentrations was developed in cooperation with the American Society for Testing Materials (ASTM),

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Committee D-19 on Industrial Water. Bromophenol blue is the indicator, and amine is determined colorimetrically. Concentrations as low as 0.1 p.p.m. are measured with reasonable accuracy. Efforts to determine concentrations of acetate in a similar range have been unsuccessful.

The laboratory boiler consists of a stainless-steel tube connected by crossovers to a second stainless-steel tube containing two electric heaters. A conventional sight glass permits examination of the steam space for carryover. Controls are provided for maintaining substantially constant steaming, blowdown, and feed-water input at any desired level. The boiler carries an ASME Code certificate and has been equipped with devices to assure the safety of employees. All heat-conducting parts of the boiler are insulated with a thick coating of special cement. Initial testing indicated that approximately 50 percent of the filming material enters the steam from the boiler water. Material balances have not disclosed what happens to the remainder of the material, since little or none is found in the blowdown.

Stability testing was begun by establishing a corrosion rate for the steam supply for the building; the Bureau's composite nipple was used to determine both weight losses and corrosion trends. Filming treatment was then added to control corrosion. In the next run the presence of an electrolyte in the steam condensate, perhaps caused by contamination or excessive carryover, virtually destroyed the effectiveness of the filming treatment. The electrolyte also apparently induced partial filming which caused serious localized corrosion in the form of pits and grooves.

Engineering Assistance

Engineering assistance was given to Federal agencies on boiler-water problems. In some instances considerable laboratory work was involved.

Close contact was maintained with the research and analytical testing of other Government and nongovernment groups through membership on committees. Cooperation was extended to ASTM Committee D-19 on Industrial Water, aimed toward the development of a special method for determining low concentrations of octadecylamine in condensate. The Bureau representative is chairman of ASME Subcommittee F, Power Test Codes Committee 19 on Instruments and Apparatus, which has completed a revision of Code Part 19.11 Determination of the Quality and Purity of Steam.

Services

The Bureau furnishes advisory boiler-water service and distributes testing equipment and reagents to Government-operated heating and powerplants.

Reports and recommendations were made on the analyses of 12 boiler-plant scales and deposits, 177 miscellaneous special water samples, and 5 boiler-water-treating chemicals. More than 13,500 water samples were analyzed, and more than 3,000 orders filled for boiler-water-testing equipment for use by Government engineers. Advisory and training services on boiler-water problems were given to various Government agencies. The number of plants being serviced increased about 1 percent during the year; the total number of boilers serviced to date is estimated at 9,600. Through the program, substantial

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savings are being realized by Government agencies in increased boiler efficiency and lower plant-maintenance and repair costs. Savings of $3,400 per year for each boiler in maintenance and replacement costs are reported by some representative plants.

Air Pollution

In connection with air-pollution work[67] a section on equipment and processes for abating air pollution from incineration was contributed to an air-pollution handbook.[68]

Combustion Characteristics of Refuse

The combustion behavior of refuse probably can be characterized by ignition temperature, burning velocity, and composition of products. These characteristics depend on the composition and bulk density of the charge, rate of airflow, ratio of undergrate to overfire air, relative motion of fuel bed and air, enthalpy of the air, percentage of excess air, and distribution of heat transfer among radiation, convection, and conduction.[69] Experiments to determine the effects of these variables are being conducted in a small cylindrical furnace arranged so that tangential, radial, and undergrate air, various combinations of these, and possibly downflow air can be supplied.

Charges consisting of wood chips, newspaper, corrugated cardboard, and leafy vegetables such as lettuce, cabbage, etc., and having moisture contents of 50 percent and 25 percent, have been used. Of five accepted methods of moisture determination, toluene distillation was adopted for reasons of speed, simplicity, and accuracy. The incinerator consists of two separable, refractory-lined parts, a gas-fired hood with an axial outlet for gaseous products, and a grate for holding the charge. To conduct a test the hood is heated to a predetermined temperature and lowered over the grate section. The air rate is adjusted, and the charge ignited by radiation from the hood. Hood, bed, and flue-gas temperatures, relative smoke density, weight of charge burned, and concentration of carbon dioxide and oxygen in the flue gas are measured at frequent intervals. Until now all tests have been made with tangential overfire air. Hood temperatures and air rates have been the principal variables. The results of these tests indicate that an increase in hood temperature is accompanied by a decrease in ignition time. Increasing the air rate has a similar effect, but beyond a certain optimum rate the reverse might be true. High air rates generally produce high burning rates.

67/ McCabe, L. C., Continued Interest in Air Pollution Control: Utilization, vol. 9, No. 2, February 1955, pp. 36-38.

68/ Johnstone, H. F., Clark, A. M., Corey, R. C., Friedlander, S. K., Lunde, K. E., Peters, M. S., Poppoff, I. G., Schneider, H. B., Semrau, K. T., and Sproull, W. T., Equipment and Processes for Abating Air Pollution: Air Pollution Handbook sec. 13 (ed. by P. L. Magill, F. R. Holden, and C. Ackley), McGraw-Hill Book Co., New York, N. Y., 1956, pp. 13-1 to 13-106.

69/ Corey, R. C., Basic Studies in Incinerator Design: Conference on Incineration, Rubbish Disposal, and Air Pollution (Air Pollution Foundation, Los Angeles, Calif.), vol. 1, No. 3, January 1955, pp. 21-24.

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Another aspect of the incineration problem is destruction of odorous effluents. The influence of time, temperature, and concentration upon the destruction of various types of odorous vapors is being measured.

Disposal of Radioactive Wastes by Incineration

Design and testing were continued of an incinerator for disposing of combustible radioactive wastes. The unit was designed to be particularly useful to hospitals, universities, and small commercial plants. Equipment for auxiliary gas firing was completed, and subsequent tests indicated that the unit operated satisfactorily. Further tests indicated the usefulness of a nozzle directed downward toward the grate of the cylindrical combustion chamber for supplying auxiliary air.

At the conclusion of the tests it was decided that combustion probably had been improved to as high a degree as possible and that further improvements, if any, would have to be in the gas-cleaning system. Accordingly, the incinerator was dismantled, shipped, and reassembled at the laboratory of the Harvard School of Public Health at Boston, Mass., where facilities for studying gas cleaning are available more readily.

Sampling of Stack Effluents

In cooperation with the U.S. Public Health Service and the Special Air Pollution Study of Louisville and Jefferson County, stack effluents from the National Carbide Co. plant near Louisville, Ky., were tested to determine their nature and quantity. A preliminary survey of the plant was made in cooperation with the various groups studying air pollution in the Louisville area, and flowsheets were prepared showing the major details of the various processes of the plant and the location of the sampling points. The major product manufactured by this plant is calcium carbide. From this it also produces large amounts of acetylene. Sampling equipment was assembled and in some instances designed to facilitate measuring the various gasflows and concentration of effluents in the stacks. Effluents were measured in six stacks, which varied from 4-1/2 to 16 feet in diameter and from 10 to 250 feet in height; and the gasflow rate, temperature, CO_2 and O_2 content, SO_2 and SO_3 content, dust concentration, and particle size were determined.

Spectrometric Analysis of Pollutants

Work on atmospheric pollution included the development of a mass-spectral method for analyzing nitrogen dioxide and other components in complex mixtures and an infrared method for measuring specific hydrocarbons in coal-mine air, pyrolytic oxidation studies of aliphatic oxygenated compounds, and detection of nitro compounds in a Los Angeles air sample.70/

Air Pollution by Sulfur Dioxide

Concentration of industry in some areas has increased awareness of the contribution made to air pollution by sulfur dioxide in flue gases from

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70/ Friedel, R. A., Spectrometric Investigations of Atmospheric Pollution: Anal. Chem., vol. 28, No. 12, 1956, pp. 1806-1810.

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powerplants. To determine the cost of decreasing the concentration of sulfur dioxide to tolerable limits, a cooperative study was undertaken with the U.S. Public Health Service.

Costs were estimated for three types of liquid scrubbing processes for removing sulfur dioxide from flue gases - the nonregenerative limestone process, the Fulham-Simon-Carves ammoniacal-liquid process, and the regenerative sodium sulfite process (Johnstone and Singh). The scrubbing plants were to handle 20 million c.f.h. of flue gases from a powerplant of 120,000 kw. capacity. The sulfur dioxide contents of the gas were 0.083 and 0.30 volume-percent, which represent the lower and upper limits of concentrations resulting from the combustion of most coals now being used. Preliminary estimates were prepared for 90-percent removal of sulfur dioxide from both gases and also for 70-percent removal from the flue gas initially containing 0.30 percent sulfur dioxide.

A literature survey has been completed of other methods of removing sulfur dioxide. Bench-scale studies of the more promising methods are underway to obtain data for evaluation.

Stream Pollution

Stream-pollution research is designed to aid industry in abating waterborne pollution resulting from mining, smelting, refining, and other mineral processing. Stream pollution is minimized when there is optimum recovery of usable material, including process water, from the waste. Data are being collected on industrial wastes of mineral origin to develop, collate, and recommend methods and processes for disposing of wastes and conserving useful materials. Establishing criteria of waste discharge and determining the effects of pollution in the streams are not included in current activities.

A comprehensive report was written on the waterborne wastes of the material industries in New England and New York,71/ and a paper on opportunities for research on utilization and disposal of waterborne mineral wastes was presented at the 1956 Annual Meeting of the American Institute of Mining and Metallurgical Engineers (AIME).

CARBONIZATION AND ITS PRODUCTS

Studies were continued of the mechanism and kinetics of coal carbonization, designed to furnish fundamental data on the formation of coke and on yields and properties of other carbonization products, as well as to develop new techniques for assessing factors controlling carbonization.

Thermal Decomposition of Coal

In low-temperature carbonization the composition of the tar depends on the rank of the coal; in high-temperature carbonization, however, this is not so because of thermal cracking of the primary tar vapor. Formation of tar at

71/ Martinez, B. P., Waterborne Wastes of the Mineral Industries New England-New York Region: Bureau of Mines Rept. of Investigations 5289, 1956, 41 pp.

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low temperatures and its transformation at high temperatures, encountered as it passes through the hotter coke and when it contacts oven walls, are being investigated.

A fluidized-bed carbonizer has been used to study the formation of tar at 450° to 500° C., and a fluidized-bed thermal cracker has been used to study its alteration at higher temperatures. The two apparatuses permit unitized operation at atmospheric pressure. A mixture of 50 percent High Splint-bed coal from Harlan County, Ky., and 50 percent char has been used as feed. The bed was agitated mechanically in addition to the agitation caused by fluidization. Both steam and nitrogen were used as fluidizing medians. Bed temperature, residence time of coal particles, and superficial velocity of the fluidizing medium were varied.

Tar yields with nitrogen have not exceeded 34 gallons per ton of coal, whereas yields as high as 42.5 gallons have been obtained with steam. Small differences have been found in the quality of the tar. Except for variation of specific gravity and paraffin content of the neutral oil with temperature, these differences have not yet been related to the operating variables. The char yield ranged from 1,200 to 1,400 pounds per ton, and its volatile-matter content from 13 to 17 percent. The yield of char was related to its volatile content, which depended on bed temperature and residence time.

Carbonizing Properties of Dull and Bright Coals

Samples representing two petrographically distinct layers of coal from the Hernshaw bed, Wharton No. 2 mine, Boone County, W. Va., and a standard sample of the entire bed were carbonized at 900° C. in the 18-inch BM-AGA retort. An 8-1/2-inch layer of dull attrital coal from the lower part of the bed contained 6.4 percent ash, 0.5 percent sulfur, and 64.5 percent fixed carbon; the top 14 inches of the bed was bright-banded and contained 3.1 percent ash, 1.1 percent sulfur, and 61.5 percent fixed carbon. The agglutinating values were 2.0 for the dull coal and 6.8 for the bright. The dull coal contracted much more than the bright coal; the contraction of the sample representing the full thickness of the bed was intermediate. The coke from the dull coal was appreciably stronger than that from either the bright coal or the sample representing the entire thickness of the bed.

Carbonization of dull and bright coals blended with 7-1/2 or 15 percent pulverized coke showed that adding this inert to the dull coal lowered its coke strength progressively. The coke-making property of the bright coal was improved by blending with 7-1/2 percent coke, but adding 15 percent made the coke abradable. These results show that, although the dull coal yielded stronger coke than the bright coal, only the latter benefited by blending with inert coke breeze.

Expansion During Carbonization

The Bureau of Mines sole-heated oven was used to determine the expanding properties of those coals tested in the survey of coking coals (see below) that contained more than 65 percent dry, mineral-matter-free fixed carbon.

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These tests are of interest industrially, since expansion of coal in commercial coking may cause oven damage.

Four Kentucky coals were tested, representing the High Splint bed from Harlan County and the Lower Elkhorn, Elkhorn No. 1, and Bingham beds from Pike County. The High Splint, Lower Elkhorn, and Bingham coals may be considered safe for commercial coking because they contracted 10.9, 4.6, and 9.5 percent, respectively; the Elkhorn No. 1 should be carbonized with care because it expanded 2.5 percent.

A resample of Lower Kittanning coal from the Bird No. 3 mine, Cambria County, Pa., expanded 49.4 percent. Inasmuch as the original sample from this mine, tested about 3 years ago, expanded 61.2 percent (an exceptionally high value), this recent result shows that the expanding property may change significantly as mining progresses.

Effect of Pretreatment on Coal Carbonization

Controlled Oxidation

The effects of pretreatment of coals on their carbonizing behavior were investigated further as part of the study of the mechanism of coking. A lignite from Milam County, Tex., was oxidized by air at temperatures from about 27° to 300° C. for varying lengths of time.72/ The oxidized coal was carbonized at 500° C. in a laboratory assay unit, and the products of carbonization were measured and analyzed. The data suggest that, as lignite is progressively oxidized, tar yields are decreased and gas yields increased in a ratio approximating 1:1. Presumably tar-yielding structures in the nonoxidized coal produce principally gas after having been oxidized. Char-yielding structures of the nonoxidized coal yielded additional water of decomposition after oxidation and pyrolysis. As lignite was subjected to greater oxidation the gaseous product evolved on pyrolysis became almost entirely carbon dioxide. Tar yields decreased as oxidation increased, so the reduction in the yield of tar may be used as a qualitative measure of oxidation.

A coal from the Middle Kittanning bed, which had marginal coking properties and was rather high in moisture and oxygen, was oxidized at 300° C. The coal was oxidized by passing heated air at high velocity through a shallow fixed bed until a small-scale abrasion test indicated unsatisfactory coke formation. The Roga abrasion test indicated a rapid decrease and virtual destruction of coke structure after 8 hours of oxidation as compared with 40 hours for Pittsburgh coal. When Middle Kittanning coal was predried at 105° C., its oxidation rate decreased and its coking power was not destroyed until after 20 hours of oxidation. These effects were less pronounced when the moisture was removed by desiccation at room temperature, which indicates that thermal drying "conditioned" the coal before oxidation.

72/ Gomez, Manuel, Goodman, J. B., and Parry V. F., Effect of Oxidation on Carbonization Yields of a Texas Lignite: Bureau of Mines Rept. of Investigations 5277, 1956, 17 pp.

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Studies of controlled oxidation of Pittsburgh coal at elevated temperatures showed little direct correlation between the oxygen content of the oxidized coal and its coking properties as measured by the small-scale carbonization tests - an observation agreeing with the conclusions of other investigators. As the decrease in coking power of oxidized coal does not depend on the increase in oxygen per se, changes in constitution of the coal may be largely responsible. These changes are more pronounced above 300° C., where thermal decomposition may be responsible for the decrease in coking.

Heating in Inert Atmospheres

Illinois No. 6 coal, which previously had been shown to yield stronger coke when preheated in steam or nitrogen at temperatures ranging from 200° to 350° C., was preheated and stored 90 days in air at room temperature. Because this coal yielded coke with strength about equal to that of other preheated samples not exposed to air, it was concluded that storage for 90 days had little effect on the coke-making properties of preheated Illinois No. 6 coal.

The investigation was extended to include the following high-volatile coals: Middle Kittanning from Ohio, Lower Sunnyside from Carbon County, Utah, and Hiawatha from Carbon County, Utah. Middle Kittanning yielded stronger coke when preheated, and this improvement increased uniformly as the preheating temperature was raised through the 200° to 325° C. range.

Lower Sunnyside benefited by preheating, and the strongest coke was obtained from samples preheated to the highest temperature (325° C.). Hiawatha high-volatile B is weakly coking and ranks lower than the other coals tested in the preheating investigation: it contained appreciable amounts of resins, and a yield of about 1 percent of this material was recovered by condensing the steam effluent from the preheater. Although preheating this coal to 320° C. and carbonizing it without intermediate cooling increased the strength of the coke markedly, the improvement did not meet present standards for western blast-furnace coke. For this reason blends containing 15 percent of either low-volatile Pocahontas or high-volatile A Hernshaw, W. Va., coals were carbonized both untreated and after preheating to 320° C. The results of these blending tests show that the coke-strength indexes for both preheated blends were substantially higher than published indexes of coke used at a Utah steel plant. These data show that preheating is a means whereby metallurgical coke may be made from blends containing a major proportion of weakly coking coal.

Effect of Inerts on Coking

Carbonization of inert-containing blends has been studied in the Tuscaloosa and sole-heated ovens. To determine the effects of highly adverse operating conditions on the silica-brick heating walls of the Tuscaloosa oven, blends containing up to 40 percent by weight of iron-ore fines were tested under conditions of high flue temperature, long coking times, and without protection against leakage of air. Photographic and visual comparison of the walls before and after each test indicated little deterioration, except at

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one place adjacent to the coke-side door. At this location, air evidently leaked into the charge, which ashed several pounds of coke and caused a small amount of slag to form on the silica brick wall. In the limited number of tests conducted no deterioration could be attributed definitely to excessive temperatures, although in one test the flues were maintained at 2,600° F. Formation of significant amounts of slag at one place in the oven emphasizes that precautions must be taken to prevent oven damage due to air leakage.

An attempt was made to determine the effects of inerts on the force required to discharge the Tuscaloosa oven. The results indicated that pushing pressures, measured with a dynamometer, usually decreased when moderate amounts of inert substances were added. In further studies in the constant-width coking chamber most changes that tended to increase the size of the coke also caused a decrease in pushing pressures. Additional study with improved methods of measurement appears desirable.

Inert substances influence coking times in at least two ways: First, many inerts exert a marked influence on charge density; second, the heat capacities of most inerts differ from those of the coals with which they are blended. Because the effects of variations in charge density have been considered previously, the effects of added inerts were determined after correcting the results to a common density. Adding iron-ore fines and anthrafines in amounts up to about 15 percent had no significant influence on the times required for carbonization. Beyond this point, however, coking times increased rapidly and exceeded those for unblended coals by approximately 2 hours for 40 percent anthrafines and by 3 hours for similar additions of iron-ore fines.

The effects of a wide range of inert substances on the expansion of various coals were determined in the sole-heated oven. Effects due to 20 or 30 percent inerts can be predicted closely when the amount and apparent specific gravity of the inerts are known. Furthermore, whereas inerts having apparent specific gravities greater than approximately 1.0 tend to decrease expansion, very light inerts having apparent specific gravities around 0.3 serve to increase expansion, provided all results are corrected to a common reference density for comparison. Increased expansion caused by adding inert substances has not been reported previously.

A further study of the effects of inerts on expansion, in this instance fluidized petroleum coke, was made in the Tuscaloosa and sole-heated ovens.73/ The results were somewhat similar to those noted by previous investigators for blends containing anthrafines and indicated that significant quantities of fluid coke could be incorporated into many blends of coal with beneficial results to physical properties.

Current efforts are being directed toward predicting the fundamental effects of adding inerts. Thus, the effects of adding up to 20 or 30 percent inerts on expansion and apparent specific gravity of sole-oven cokes can be

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73/ Gayle, J. B., and Eddy, W. H., Carbonizing Tests With Tuscaloosa Oven: Use of Fluid Petroleum Coke as a Blending Agent: Bureau of Mines Rept. of Investigations 5276, 1956, 22 pp.

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predicted closely from the apparent specific gravity and quantity of the inert substance. For anthrafines smaller than 48-mesh, the effects on the 1/4-inch tumbler indexes can be predicted for certain strongly coking coals.

Effects of Flue Temperature and Bulk Density on Coking

During the year, emphasis was placed upon determining the effects of flue temperature on coking time.74/ The end of the coking period was defined in terms of temperatures measured at the center of the charge. Coking times for the Tuscaloosa oven varied as the 2.40 power of the flue temperature (expressed in degrees Rankine); no allowance was made for soaking or tempering the charge. A similar analysis of published data for commercial ovens yielded a value of 2.66, which agrees closely with the experimental result. Incidental determination indicated that coking times also vary as the 1.08 power of bulk density. This confirms the results of an earlier study, which showed that change in bulk density causes an approximately equal percentage change in coking time.

In tests of 11 coals and 6 blends, each tested at several bulk densities, yields of coke, gas, and ammonia and the heating value of the gas were independent of bulk density within the range covered.75/ Yields of tar increased and those of light oil decreased slightly as density increased. Apparent specific gravity, stability, and hardness of the coke, as well as the expansion force in the slot-type oven, increased as density increased, whereas the shatter index decreased.

Effect of Fluidity on Abradability of Coke

As reported previously, the abradability of chars obtained from blends of coke breeze and Pittsburgh-bed coal varied with size of coke and composition of blend. A possible explanation involves the theory of foam stability and assumes that plastic coal has come of the properties of foam. When the bubble structure, or foam, of plastic coal is stabilized and persists during carbonization, the resulting solid residue has high porosity and relatively thin walls; both of these characteristics may favor easy abradability. When the bubble structure has low stability, the cell walls tend to collapse before solidification, and the resulting char is more dense and less abradable. Size of the inert well may be a controlling factor. An optimum coke size, intermediate between the finest and largest used in these tests, increased the foam stability most effectively.

The fluidity study was extended to include blends containing a low-porosity inert material in a wide range of sizes. The abradability of the chars made from coal-silicon carbide blends varied with the particle size of this inert and also with the blending ratio. The strength of the char,

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74/ Gayle, J. B., and Eddy, W. H., Carbonizing Tests With Tuscaloosa (Ala.) Oven: Dependence of Coking time on Flue Temperature: Bureau of Mines Rept. of Investigations 5186, 1956, 20 pp.

75/ Reynolds, D. A., Wolfson, D. E., Smith, F. W., Coal Carbonization. Relation of Charge Density of Coal to Carbonizing Behavior. Proc. Blast Furnace, Coke Oven, and Raw Materials Committee, Iron and Steel Div., AIME, 1955, vol. 14, pp. 224-242.

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as indicated by the abrasion index, increased as the particle size of the inert decreased. Apparently this can be ascribed to more points of contact with the fluid substance in the coal that makes the char more coherent and less abradable. The abradability of chars made from coal-silicon carbide blends did not show the unusual behavior exhibited by chars made from blends containing the coarser sizes of coke breeze. As the proportion of coal in the blend was increased, the abrasion index increased rapidly until it equaled the value for char from the unblended coal. Further increase in the coal-silicon carbide ratio caused no appreciable change in the index. This behavior was somewhat similar to that shown by blends containing coke breeze of the finest size.

Correlation of BM-AGA Test With Commercial Carbonization

The BM-AGA carbonization test was correlated with commercial practice in cooperation with the Canadian Department of Mines and Technical Services. Samples of coal blends being coked at three Canadian steel plants were carbonized at 900° C. in both the 18- and 13-inch retorts. The physical properties of the experimental and commercial cokes were determined by the same standard methods - ASTM apparent specific gravity, shatter, and tumbler tests. The 1-1/2-inch shatter, 1-inch tumbler, and 1/4-inch tumbler indexes of the experimental (18-inch retort) and commercial cokes from the two strongly coking blends were very similar. Agreement of the 1/4-inch tumbler and 1-1/2-inch shatter indexes likewise was close for the third blend, which yielded weaker coke. Only the 1-inch tumbler indexes for the third blend, which yielded the weakest coke, differed significantly. In this instance the low result was obtained for the commercial coke; it may be due partly to a higher percentage of moisture in the coking blend, which would lower the charge density.

Coking Properties of Western Coals

A study was initiated to establish the coking properties of several western coking coals and blends by carbonization under comparable and specified conditions; to determine the petrographic distribution in the whole coals, selected sizes, and blends; and to promote standardization of techniques for measuring coking properties. The study is being conducted in cooperation with the Columbia-Geneva Steel Division of the United States Steel Corp., the Kaiser Steel Corp., The Colorado Fuel and Iron Corp., and the Colorado School of Mines Research Foundation. The program includes studies of the effect of size, bulk density, temperature, addition of char, and addition of oil on coking properties. Preliminary tests were made on four western coking coals: Geneva mine, Sunnyside seam, Carbon County, Utah; Sunnyside No. 1 mine, Lower Sunnyside seam, Carbon County, Utah; Allen mine, No. 1 seam, Las Animas County, Colo.; and Hawk's Nest mine, "E" seam, Gunnison County, Colo.

Nine carbonization tests were made in the 500-pound slot-type oven. Four single tests were made on representative samples of the four coals; one coal was tested in duplicate to establish reproducibility of the coke oven. Additional tests were made on the Kaiser Steel Corp. coke-plant mix at 1,980°, 2,080°, and 2,280° F. The physical properties of coke made in the Kaiser ovens agree closely with those of coke made in the 500-pound coke oven at the

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corresponding temperature and conditions of carbonization. A total of 49 carbonization tests were made on the 4 representative samples in a 50-pound, 10-inch circular retort. Adding up to 15 percent char to Sunnyside No. 1 and Allen coals increased the stability of coke. Significant improvement in the quality of the coke occurred when 15 percent char was added to Geneva coal. Hawk's Nest coal, under the conditions tested, produced a coke susceptible to considerable degradation from handling, drop shatter, and tumbling. Carbonizing Hawk's Nest coal with 15 percent low-temperature char increased the average size and the coke-strength index.

A small-scale carbonization test was developed, suitable for rapidly evaluating many blends, sized fractions, and relatively pure petrographic samples available only in small quantity. This 100-gram test is highly reproducible, and the coke obtained appears to bear a relation to coke made in the 500-pound slot oven and the 50-pound, 10-inch circular retort. An investigation was begun with the primary objective of relating the bench scale and laboratory slot-oven carbonization tests.

Samples of each of the four coals were submitted for petrographic analysis to the Steinkohlenbergbauverein laboratories in Essen, Germany. The samples were analyzed as approximately 1/3 "vitrit," 1/2 "clarit," and 1/3 intermediate grades; pure "fusit" and "durit" appeared to be minimal. (The German terms are used here because exact English equivalents are lacking.) Preliminary studies suggest that the vitrit-to-clarit ratio may have some significance in relation to the coking property of western coals. A series of sized coal fractions was prepared from the four coals studied. These samples were shipped to the Steinkohlenbergbauverein laboratories for petrographic analyses.

Carbonizing Properties of Bituminous Coals

Additional reports from the survey of the minable reserves of coking coals in the United States were issued during the year.[76 77 78 79 80 81]

76/ Dowd, J. J., Provost, J. M., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Marshall County, W. Va.: Bureau of Mines Rept. of Investigations 5207, 1956, 21 pp.

77/ Hershey, R. E., Williams, Lloyd, Crentz, W. L., Miller, J. W., and Reynolds, D. A., Estimate of Known Recoverable Reserves and Preparation and Carbonizing Properties of Coking Coal in Claiborne County, Tenn.: Bureau of Mines Rept. of Investigations 5229, 1956, 44 pp.

78/ Blaylock, D. W., Dowd, J. J., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Clarion County, Pa.: Bureau of Mines Rept. of Investigations 5231, 1956, 27 pp.

79/ Travis, R. G., Dowd, J. J., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Monongalia County, W. Va.: Bureau of Mines Rept. of Investigations 5233, 1956, 30 pp.

80/ Williams, Lloyd, Gibbs, H. K., Crentz, W. L., Miller, J. W., and Reynolds, D. A., Estimate of Known Recoverable Reserves and Preparation and Carbonizing Properties of Coking Coal in Campbell County, Tenn.: Bureau of Mines Rept. of Investigations 5258, 1956, 78 pp.

81/ Provost, J. M., Dowd, J. J., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Marion County, W. Va.: Bureau of Mines Rept. of Investigations 5267, 1956, 30 pp.

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As reserves of coals currently used to produce metallurgical coke become depleted, this evaluation will provide data on reserves and properties of substitute coals not previously used for this purpose. It will be valuable in time of national emergency, when demands on the steel and coal industries are great.

To complete the county reports on carbonizing properties of coals of the United States, samples were taken in areas of significant minable reserves that had not been tested. Carbonization tests by the BM-AGA method were made at 900° C. on 17 coals from Kentucky, 6 from West Virginia, and 3 from Pennsylvania. All ranked as high-volatile A bituminous coals.

Floyd County, Ky.

Three samples from different beds contained 57.8 to 59.8 percent fixed carbon on the dry, mineral-matter-free basis. Elkhorn No. 1 from the Betsy Lane No. 1 mine was low in ash and sulfur and yielded rather weak coke. Elkhorn No. 2 from Lawson No. 2 mine was low in ash but contained 1.5 percent sulfur; its coke was moderately strong. Fire-clay coal from Powell Branch No. 1 mine contained 11.2 percent ash and yielded weak coke; its fixed-carbon content (57.8 percent) is low for metallurgical use.

Harlan County, Ky.

A report has been published on the carbonizing properties of Harlan County coals.82/ Twenty samples from eleven beds were carbonized singly. To determine the effect of blending coals from this county with higher-rank coal from West Virginia, seven coals were blended with various proportions of low-volatile coal. The Harlan County coals differed significantly in coke-making properties when carbonized singly. The 1-1/2-inch shatter index ranged from 18 to 81; ranges of 1- and 1/4-inch tumbler indexes were 6 to 39 and 43 to 67, respectively. Blends of the weakly coking Harlan County coals with low-volatile coal coked fully as strongly as the equivalent blends of Pittsburgh district coals that yielded stronger coke when carbonized singly.

Knott County, Ky.

Elkhorn No. 3 from Bradley No. 1 mine and Elkhorn No. 2 from Bradley No. 2 mine were low in ash, but both contained 1.6 percent sulfur. Because their cokes were rather weak, these coals probably would not be used in coking blends if more strongly coking coals, such as the Elkhorn coals of Pike County, were available.

Letcher County, Ky.

Hazard No. 4 coal from Sizemore No. 2 mine contained moderate proportions of ash and sulfur and coked strongly enough to qualify for use in metallurgical blends. Elkhorn No. 3 was low in ash but contained 1.8 percent sulfur. Because its coke was rather weak, this coal is not considered a first-class metallurgical coal.

82/ Wolfson, D. E., and Birge, G. W., Carbonizing Properties of Harlan County, Ky., Coals: Bureau of Mines Rept. of Investigations 5257, 1956, 17 pp.

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Pike County, Ky.

Lower Elkhorn-bed coals from the Feds Creek and Ratliff mines, Elkhorn No. 1 from the E. Mullens No. 2 mine, Elkhorn No. 2 from the Allegheny No. 4 mine, and Bingham from the D. J. B. mine were low in both ash and sulfur, and all coked as strongly as the average high-volatile coking coal now used by the steel industry. These five coals ranked higher than the other Kentucky coals tested this year; they contained 62.6 to 66.0 percent fixed carbon on the dry, mineral-matter-free basis. Another sample from the Bingham bed, which represented the Potter No. 4 mine, yielded rather abradable coke, but this coal probably would be benefited by cleaning, as it contained 10.3 percent ash. A second sample of Elkhorn No. 2 coal from the Elkhorn No. 2 mine contained 59.6 percent fixed carbon and yielded weaker coke than coal from the Allegheny No. 4 mine in the same bed. Two samples from the Elkhorn No. 3 bed, Bear Fork No. 3 and K-H No. 2 mines, were very low in ash and yielded cokes with intermediate-strength indexes. Haddix coal from Cinderella No. 2 mine was low in ash and sulfur but yielded weaker coke than the other samples from this State. It contained only 58.2 percent fixed carbon.

Greene County, Pa.

Three Pennsylvania coals were from Greene County. Sewickley from Luxener mine yielded weak coke, but the sample was taken from a strip mine and may have been weathered. Another sample from this bed, representing Whitely No. 3 mine, yielded strong coke, and it should be suitable for metallurgical use if its contents of ash and sulfur (10.4 and 1.3 percent, respectively) are lowered by cleaning.

Waynesburg from Hartley mine contained 15.8 percent ash and 2.0 percent sulfur. If this coal is benefited appreciably by cleaning, it should be suitable for metallurgical use.

Somerset County, Pa.

A report has been published on the carbonizing properties of Somerset County coals.83/ Two samples from the Upper and Lower Freeport beds were carbonized in this investigation. The coals described in this report rank as low-volatile bituminous, as do most Somerset County coals. All samples were carbonized as binary blends, with Pittsburgh coal as the major constituent. Low-volatile coals are not coked singly in modern ovens because they expand excessively. This report shows that low-volatile coals from the four major beds in Somerset County are satisfactory metallurgical blending coals. Each of the four Somerset County coals was substituted for Pocahontas No. 3 in blends with high-volatile A Pittsburgh coal without significant loss in coke strength. Although expansion of the Somerset County coals, as determined in the sole-heated oven, ranged from 11.5 to 61.6 percent at a bulk density of 55.0 p.c.f., expansion of the equivalent 10-, 20-, and 40-percent

83/ Wolfson, D. E., Birge, G. W., and Naugle, B. W., Carbonizing Properties of Somerset County, Pa., Coals: Bureau of Mines Rept. of Investigations 5269, 1956, 19 pp.

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Somerset County-Pittsburgh and Pocahontas No. 3-Pittsburgh coal blends were similar. The expanding property of a low-volatile coal generally is not closely related to the expansion of its blend with a high proportion of high-volatile coal; the expansion of such blends depends primarily on the expanding property of the high-volatile coal.

Boone County, W. Va.

Six West Virginia coals represented six beds in Boone County. They contained 59.5 to 63.8 percent dry, mineral-matter-free fixed carbon; therefore, they do not rank high in the high-volatile A classification.

Dorothy coal from Dorothy No. 3 mine and No. 5 Block from No. 1 mine contained moderate proportions of ash and were low in sulfur. As both yielded strong coke, they should be entirely suitable for metallurgical coking blends. Stockton-Lewiston from No. 2 mine yielded abradable coke, but this coke probably would be suitable for metallurgical use if its high ash content (13.2 percent) were lowered. No. 2 Gas coal from Shelby Jean Stone mine and Cedar Grove from Ziegler and Vance No. 2 mines yielded moderately strong coke, but the former contained 2.3 percent sulfur, which would detract from its value for metallurgical use.

Hernshaw coal from Wharton No. 2 mine contained moderate proportions of ash and sulfur and has satisfactory coking properties. Layer samples from this mine were tested separately to determine the carbonizing properties of petrographically different constituents.

Metallurgical Use of Anthracite

Despite the metallurgical use of anthracite as a fuel in blast furnaces and foundry cupolas in the latter half of the 19th century, its use has declined to a point where, except as a minor blending component in the production of coke, virtually no major tonnages of anthracite are used in present-day metal-melting and -reducing processes. Yet the location of anthracite deposits with respect to major steel-producing areas and the industrial northeast, makes their use attractive from a transportation viewpoint. Furthermore, a 300-year supply is indicated at a production rate of 30 million tons per year.

Anthracite appears to possess most of the characteristics of a satisfactory metallurgical fuel, except for its resistance to impact and abrasion, as indicated by the ASTM Tumbler Test for Coke and its lack of size stability when subjected to thermal shock. Preparing an agglomerated fuel as resistant to impact and abrasion as metallurgical coke and also stable to thermal shock has been investigated. Thermal stabilization of lump anthracite and its use in foundry cupolas also have been studied.

Internal stresses in anthracite lumps are believed to result from many factors in the constitution and formation of anthracite deposits. Among these are layer-on-layer accumulation of the original organic materials, immense horizontal and vertical pressures applied during coalification,

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organic remnants that decompose to give volatile gases at higher temperatures, evolution of inherent moisture, and characteristics of the noncombustibles intermixed with the carbonaceous materials. The idea of relieving these stresses by preheating the fuel at controlled rates and temperatures is not new, but most experimental results have had to be interpreted from small-scale batches and laboratory tests that could not be closely related to industrial processes. To obtain closer correlation, 75 tons of anthracite, comprising three standard sizes, were calcined in a continuous, pilot-size retort; and the lump product from the egg-size feed was tested as a fuel in a commercial iron-melting foundry cupola. All five runs with calcined anthracite as 100-percent cupola fuel, both in the initial bed and with subsequent metal charges, gave the hot-metal temperatures and output regularly required in this foundry.

Blending and Cupola Fuel

In recent years economic factors have provided considerable incentive for establishing new markets for anthracite, and the success with which some companies have incorporated moderate quantities of anthrafines into coking blends has led to widespread interest in the possibility of extending this practice.

To obtain additional information on this subject several blends containing up to 40 percent anthrafines were carbonized in the Tuscaloosa oven, and the properties of their cokes determined by standard physical tests.84/ The results were quite varied, and in some instances opposing trends were noted when the composition of the base blend or the size of the anthrafines was changed. Although general conclusions on the effects of adding anthrafines on the physical properties of metallurgical cokes were not possible, the variety of results obtained by varying the size and quantity of anthrafines according to the composition of the base-coal blend showed that the procedure is a versatile method for tailoring coke to desired specifications.

An investigation was made of an agglomerated metallurgical fuel, predominantly anthracite, with various binders, which, after calcination at elevated temperatures will equal or be superior to good coke in resistance to impact and abrasion.

Initial experiments to determine the type of equipment most suitable for forming briquets included tests with auger extruders, hydraulic extruders, roll presses, and low-temperature Disco-type equipment. The experiments indicated that roll pressing and auger extruding gave the most satisfactory quality. Low-temperature, drum-forming techniques with bituminous coal as the binder did not produce briquets of the desired strength or density, and the yield of plus-1-inch product was only about 23 percent of the total yield. Figure 11 is a photograph showing the porous structure of one of the larger pieces of agglomerate containing 45 percent anthracite and 55 percent bituminous coal. The tests conducted with hydraulic extruders were not promising enough to warrant buying equipment of this type.

84/ Gayle, J. B., Eddy, W. H., and Brown, R. L., Use of Anthrafines for Producing Metallurgical Coke: Bureau of Mines Rept. of Investigations 5287, 1956, 19 pp.

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FIGURE 11. - Anthracite-Bituminous Agglomerate Using Low-Temperature Drum-Forming Techniques. (Briquet size approx. 5 inches diameter).

Following the initial study of equipment, bench-scale experiments were made on a laboratory briquetting press and a gas-fired calcining furnace to determine the combination of raw material and calcination procedures that would produce the briquet most resistant to impact and abrasion. Coal-tar and petroleum pitches as well as bituminous coals were used as binders. Briquet-forming pressures and calcination temperatures were investigated over a wide range of conditions. The data indicate that a strong agglomerated briquet can be formed from anthrafines with 8 to 12 percent low-melting-point coal-tar pitch as the binder and 10 percent moderately coking bituminous coal at forming pressures of 2,000 to 3,000 p.s.i. After calcination at 1,750° F., briquets containing 10 or 12 percent pitch are more resistant to impact and abrasion than the foundry coke being used in metallurgical operations.

Pilot-scale briquetting equipment has been procured, including an auger-extrusion press, a roll press, and a vertical-shaft calcining furnace. An integrated pilot plant, utilizing common feeding and mixing equipment for the briquetting presses, has been designed and construction begun. The plant will allow a detailed study of process economics, materials handling, and production of tonnage quantities of briquets for testing in full-scale metallurgical equipment.

Calcination

Anthracite was calcined in the vertical, externally heated retort shown in figure 12. The retort, built of refractory shapes, has a rectangular cross section of 3 feet by 1 foot inside and is 12 feet high, with charging and filling hoppers above

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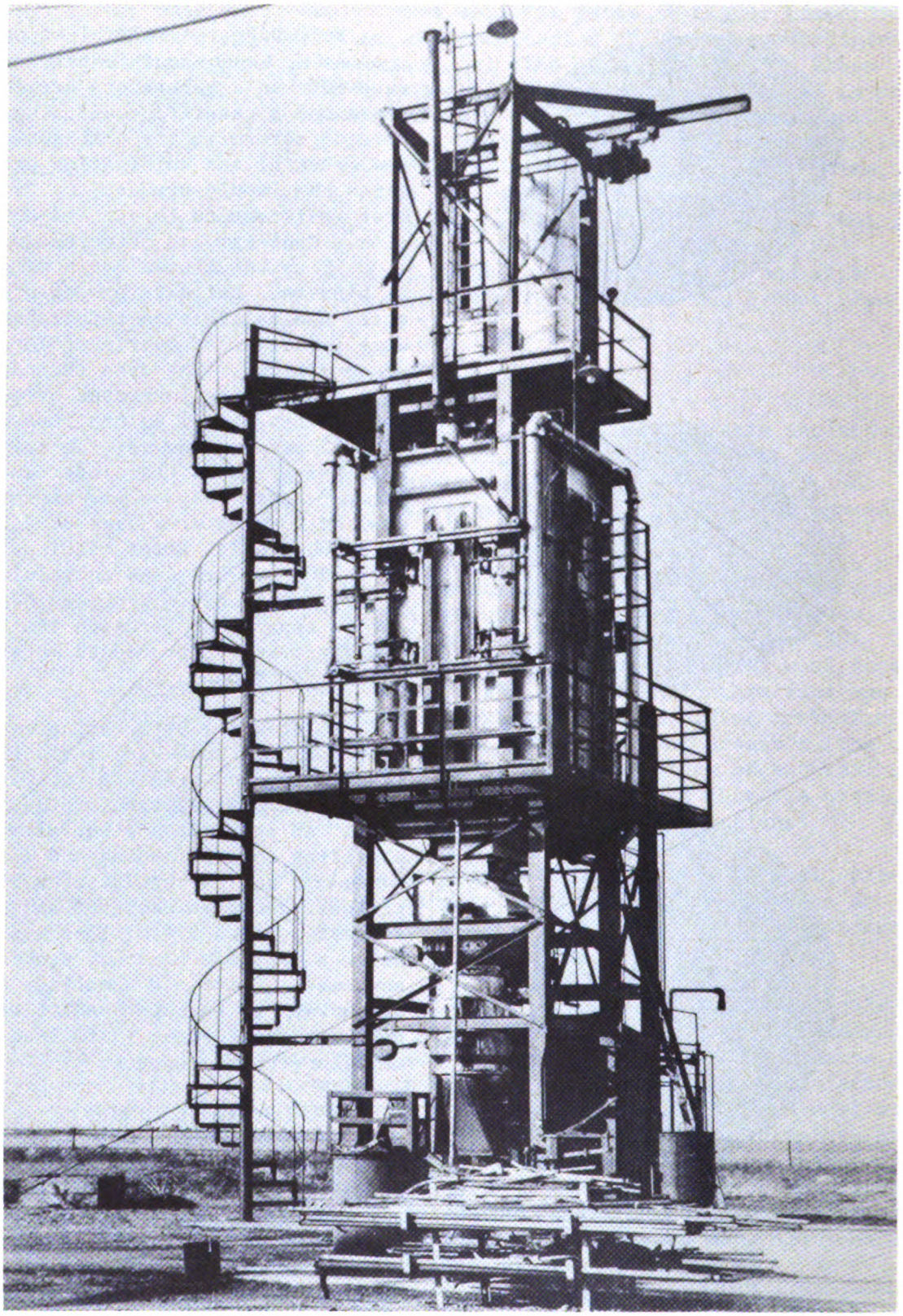

FIGURE 12. - Bureau of Mines Pilot-Scale Continuous Shaft Calciner.

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and cooling and discharging mechanism below. The discharge mechanism could be run at various speeds to change the rate at which the anthracite moved downward. The retort was heated by gas burners in horizontal flues, one above the other on the longer sides. The fuel input to each burner could be regulated independently to set up the temperature gradient from top to bottom as desired for each run. The maximum flue temperature of 2,400° F. and coal rates from 50 to 200 p.s.f.h. of retort section were tried in this series of runs. Anthracite was preheated on its way down to the retort by volatile gases and moisture, driven off from the coal in the warmer zones and passing upward to the vent just below the charge hopper. Temperatures at the top of the retort were varied from 400° to 1,500° F.; at the higher temperatures heat was carried up into the preheating zone.

Three standard sizes of anthracite were processed: Buckwheat No. 1 and Chestnut were treated in two runs each to obtain data on the design of the apparatus. Egg size was tested in 10 runs for similar reasons; also to obtain information on the effect of temperature gradients on the product and for a supply of calcined product for full-scale cupola testing. Egg-size anthracite yielded the best product when the top flue temperature was held to a maximum of 400° F., the bottom temperature at a maximum of 1,650° F., and throughput at the rate of 50 p.s.f.h. Some 40 percent of the egg lumps were recovered in this size range compared with 30 percent from egg anthracite treated at higher temperatures and faster rates. The average loss in weight from feed to product was about 10 percent, presumably mostly moisture and the 7 percent volatile matter in the raw coal.

The smaller sizes showed less degradation even when treated at higher temperatures. Buckwheat No. 1 retained 75 percent above the minimum standard size when calcined to 0.5 percent volatile matter and 79 percent when reduced to 2 percent volatile matter. Chestnut retained 66 percent above minimum standard size at 0.5 percent volatile matter and 72 percent when reduced to 2 percent volatile matter.

Egg-size anthracite from all continuous runs was tested for its resistance to impact and abrasion and drop-shatter resistance. In each instance resistance was lower than that of the original anthracite and much lower than that of metallurgical coke; however, these tests, although used to indicate the suitability of coke for metallurgical operation, actually measure handling characteristics and give no insight into the behavior of the fuel in the stock column, except with regard to abradability and load-carrying ability. Since the greatest limitation in the use of anthracite may be thermal rather than mechanical decrepitation, the calcined material was tested in a cupola.

Pilot-plant calcination of anthracite produced a supply of lump fuel for the commercial cupola tests (discussed below) and a quantity of calcined material from the Chestnut and Buckwheat No. 1 sizes and the fine screenings. The screenings were removed from the sized coal, put through the calciner, and are available for further study of anthracite fines for industrial purposes, either directly or in some agglomerated form. Observations indicated some modification of furnace design would be necessary for more satisfactory operation with anthracite. Of particular importance is the necessity for better

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regulation and control of the early heating rate in the range 500° to 1,000° F., increased cooling capacity to assure that the product is discharged from the apparatus at well below ignition temperature, and inert atmosphere throughout the processing chambers to eliminate the possibility of combustion of the coal at any stage as well as possible internal explosions of coal gases because of air leakage.

Testing in an Iron-Melting Foundry Cupola

Plant tests in an iron-melting foundry cupola were made with the calcined product from egg-size anthracite in the operating foundry at the repair shops of a large anthracite producer. The cupola is the Colliau type, lined down to a nominal inside hearth diameter of 48 inches, and having a shaft height of 109 inches from tuyeres to charging-door sill. Airblast was supplied at atmospheric temperature by a positive rotary blower with power to deliver at pressures above 32 ounces. The cupola was scheduled to operate every fourth working day and melt up to 15 tons of iron per run at a rate of 7-1/2 tons per hour. Metal was tapped intermittently; slag was retained until the hearth bottom was dropped at the end of the run.

The regular fuel charge to the cupola for several years had consisted of 60 percent raw egg-size anthracite and 40 percent of plus-6-inch foundry coke in both the initial bed and with the charges. The anthracite contained 5 to 6 percent volatile matter and 10 to 11 percent ash and had an exceptionally high stability against abrasion when subjected to the tumbler tests. Two runs with this mixture of fuels were observed, and special attention was given to the airblast pressure drop through the stock column to the temperatures of the molten iron as it was tapped.

Calcined anthracite was used in six runs. In the first the fuel comprised 60 percent calcined anthracite and 40 percent coke; in the others the initial bed and all subsequent fuel charges were 100 percent calcined anthracite. This anthracite was screened before charging but only to remove pieces under 1-5/8-inch (round hole), and so contained some 60 percent under the egg-size minimum. Substituting this calcined product for the 60 percent raw anthracite reduced the average blast-pressure drop slightly and gave a slightly higher iron temperature. All five runs with 100 percent calcined coal had maximum pressure drops well below the capacity of the lower and gave iron temperatures averaging 50° to 75° F. higher than the raw coal-coke mixture. Melting rates and outputs remained substantially the same. No other operating differences were encountered beyond a slightly longer time to get the initial bed heated by natural draft.

For comparative purposes two runs were made with 100 percent foundry coke as fuel and one run with 100 percent raw egg anthracite. The coke (all 6-inch and over) opened the stock column to give very low blast-pressure drops and greatly increased flue-gas temperatures above the column but showed no difference in the temperatures of the molten iron as tapped. With 100 percent raw coal, the pressure drop was so high that the air input had to be reduced, and the temperatures of molten iron averaged more than 100° lower.

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Other effects of changes in cupola fuel, especially on the chemical constitution and strength of the iron tapped, will be available later from tests of sample bars cast at intervals during each run; however, calcined anthracite of 1-5/8-inch size and larger is suitable as the sole fuel in cupola operations at one plant. An indispensable feature of this operation was the use of an air blower to maintain the specified rate of air at the higher pressure required.

Comparison of the results of tests with calcined anthracite and those with coke or coke-raw anthracite mixtures does not show the differences that could be expected if the tumbler and drop-shatter tests were satisfactory indicators of anthracite performance in cupola operations. Higher bustle pressures than those obtained with coke were anticipated because of the relatively small size of the calcined anthracite; however, these pressures did not increase appreciably as the melts progressed, and indications were that the runs could have been continued had the demand for metal existed. Thus the characteristic in anthracite most important for metallurgical purposes is its tendency to decrepitate. This characteristic can apparently be controlled by calcining raw anthracite between 1,880° and 2,400° F.

Low-Temperature Carbonization

Carbonizing Uraniferous Lignites

Organic volatile matter and the nature of the carbon in uraniferous lignite may interfere with the direct extraction of uranium, as neither the acid nor the alkaline leach can be filtered properly owing to the formation of gels. Moisture and some of the carbon and organic volatile matter must be removed from raw lignite before U_3O_8 can be extracted. Thus uraniferous lignite must be heated under controlled conditions to remove some of the carbon and part of the volatile organic matter; the temperature of treatment should be between 450° and 600° C. to avoid conversion of uranium to insoluble forms.

A series of tests was made of samples of uraniferous lignite from the Slim Buttes, Cavehill Mining Co. claim, Harding County, S. Dak., to study the effect of progressively increasing temperatures on fixed-bed carbonization of lignite. The temperature in seven duplicate tests ranged from 200° to 600° C. Weight loss of the moisture-free lignite was 0.5 to 20.0 percent over the temperature range. At all temperatures gas was the major product of decomposition; its heating value varied from 38 to 128 B.t.u. per cubic foot. The ash content of the soft, friable char was raised from 61.6 percent (moisture-free basis) in the original sample to 72.5 percent after heating to 600° C. The 400° C. char contained 67.7 percent ash. Very little tar or light oil was given off by this lignite; the maximum was 1.6 percent of the m.a.f. material when heated to 600° C.

For treating Manidon Mining Co. uraniferous lignite, obtained from the Edgemont, S. Dak., stockpile, internal heating in a retort was applied. Owing to the temperature limitation imposed on the pilot plant because of the high-ash chars, the dried lignite had to be carbonized in four stages; char from the first stage was the feed for subsequent stages. The later stages were carried out with internal heating only, using limited amounts of air in the

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lignite-transport gas system for partial combustion of the char; the temperature was kept low enough to avoid chemical modifications of the uranium minerals. The best approach to this problem appears to be controlled combustion of the pulverized lignite and char with air diluted by inert combustion gases to retard the rate of combustion and lower the temperature of the flame. Conventional thick-bed combustion does not appear to be practical because of the difficulty of dissipating heat rapidly enough to prevent high temperatures.

Drying and Carbonizing Fine Coal

Studies of fluidized and entrained drying and of carbonization of low-rank noncoking coals have been continued. The purpose of this investigation is to make char for producing electric power and tar from which chemicals may be extracted.

A report has been published on pilot-plant applications of these techniques to lignites from Texas and North Dakota and coals from Colorado, Utah, Wyoming, and the Philippine Islands.85/ Comparison of tar yields obtained from carbonizing several coals by the low-temperature assay and in the pilot plant showed close agreement. Pilot-plant tests on Texas lignites at 900° to 1,100° F. showed that maximum tar yields were obtained at 950° F., probably the optimum temperature for commercial carbonization of this lignite. The excess low-B.t.u. gas obtained at higher temperatures can be considered to have fuel value only. Unless a better use can be developed for such gas (130 to 280 B.t.u. per cubic foot), the most economical carbonization conditions are those yielding a maximum amount of char.

The economics of processing are closely related to the quantity and quality of the tar recovered. Samples of the tars have been submitted to various industrial and research organizations for evaluation.

Carbonizing Noncoking Coals at Elevated Pressures

Small-scale carbonization in either hydrogen or nitrogen atmosphere included the use of steam-dried lignite and Wyoming subbituminous coal in addition to the as-mined lignite previously tested. The principal objectives of these tests were determination of yields and physical as well as chemical characteristics of products pyrolyzed at 935° F. and different pressures. Such data also indicate the effect of carbonization products on gasification in the updraft pressure gasifier and possible operating troubles due to agglomeration of solid residue.

In all instances the yield of solid residue (based upon m.a.f. coal) decreased and that of gas increased with higher pressure due to increased hydrogenation. Yields of tar oils from lignites also increased with pressure, but tar yield from subbituminous coal was lowest at 400 p.s.i.g. No trend was apparent for yields of water of decomposition. Lignite, either as received or steam dried, was hydrogenated more readily with increasing

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85/ Parry, V. F., Landers, W. S., and Wagner, E. O., Low-Temperature Carbonization of Lignite and Noncoking Coals in the Entrained State: Min. Eng., vol. 8, No. 1, January 1956, pp. 54-64.

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pressure than was subbituminous coal. None of the coals agglomerated at 400 p.s.i.g., the pressure considered for operating a gasifier for lump coal, although slight agglomeration occurred at higher pressures of hydrogen. Modifications in operating procedure, including the addition of lignite tar before carbonization and charging into a preheated carbonizer, did not increase the agglomeration tendencies of the solid residue.

Carbonization-Assay Survey

The results of a survey of some North Central U.S. lignites were published during 1956.86/ The bench-scale carbonization survey covered samples of lignite from all major producers in North Dakota and a large number of smaller mines, as well as cores from drilling operations, made available through the cooperation of the Great Northern Railway.

Utilization of Lignite From India

High-moisture, low-rank lignite from South Arcot, India, represents an important source of energy in the industrial development of South India. If solid fuels, byproducts, and electric power become readily available in this area, they can contribute greatly to the rapid development of the region.

The Bureau of Mines was asked by ICA to cooperate in a study of the properties of Neyveli lignite and to suggest schemes of utilization. The large sample tested, containing 56 percent moisture and having a heating value of 4,900 B.t.u. per pound, was easily dried in the entrained-bed dryer to a product containing 2.5 percent moisture and having a heating value of 11,000 B.t.u. per pound. Subsequent fluidized carbonization yielded 1,300 pounds of char having a heating value of 12,170 B.t.u. per pound and 25 gallons of tar plus light oil per ton of dried lignite.

While in India surveying the utilization problems in South Arcot, a plan of development was recommended, including American methods of preparing and utilizing lignite. This plan calls for an orderly development of the facilities at Neyveli over a period of about 12 years.

To implement the initial phase of this plan a pilot plant capable of drying 2.5 tons of raw lignite per hour, a 1-ton-per-hour briquetting press, and a 500-pound-per-hour briquet carbonizer with associated tar train is to be provided from ICA funds for use at Neyveli. The contract for constructing this equipment and for procuring laboratory equipment has been awarded. Additional research will be conducted at Denver on a 10-ton sample of Neyveli lignite to establish optimum conditions for briquetting and carbonizing. The pilot plant should be fabricated and ready for shipment by August 1957 and installed in India by January 1, 1958.

86/ Hoeppner, J. J., Opland, M. L., and Fowles, W. W., Carbonization Characteristics of Some North-Central United States Lignites: Bureau of Mines Rept. of Investigations 5260, 1956, 21 pp.

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Low-Temperature Tar

Yield and Composition of Tar as Functions of Rank of Coal and Oxidation

Recent developments in low-temperature coal carbonization, especially fluid-bed and entrainment techniques, have resulted in renewed interest in low-temperature coal tars. Tars produced by these techniques in internally heated reactors are more primary in nature than those produced by high-temperature processes.

In the newer processes the composition of the tars and oils is dependent on that of the coals carbonized. A report was prepared on the oxidation of tar[87/] produced in pilot-plant low-temperature carbonization of coals.

The differences in composition of low-temperature tars produced under similar conditions from coals of different rank are shown graphically in figure 13. Tar yield was a linear function of the B.t.u. content of volatile matter in the parent coal, which can be related to rank of coal.

Tar From Lignite

Work on the products of low-temperature carbonization of lignite is being directed toward a qualitative examination of one commercial tar to establish procedures for investigating tars obtained from other sources or under varying conditions of carbonization and recovery.

Experimental work on this tar has continued to center largely on the tar acid fraction first recovered by conventional alkali extraction. More recently, countercurrent liquid-liquid extraction was applied, using aqueous methanol and mixed hexanes as the solvent phases. The product was more stable thermally and of equivalent purity; 23 percent of the acid boiled below 220° C. (15 percent between 200° and 220° C.) and 50 percent above 270° C. A wide variety of alkyl-substituted hydroxy aromatics and minor amounts of alkoxyl homologs were present. Where possible pure phenolic compounds were separated and characterized by physical properties and/or chemical derivatives. The majority of compounds, however, was identified by comparing infrared spectra with those of known compounds. To date the spectra of 67 phenols have been obtained in the 7-1/2- to 15-micron range.

Some work was done on neutral oil from acid-alkali extraction of tar. Exhaustive extraction of the whole oil with sulfuric acid removed approximately 70 percent of the material but did not entirely eliminate oxygen as a constituent of the components of the oil. Distillation procedures have separated in relatively pure form materials indicative of long-chain normal paraffins, and anthracene was separated from the aromatic portion.

87/ Gomez, Manuel, Goodman, John B., and Parry, V. F., Changes Induced in Low-Temperature Tar by Oxidation and Storage: Bureau of Mines Rept. of Investigations 5227, 1956, 12 pp.

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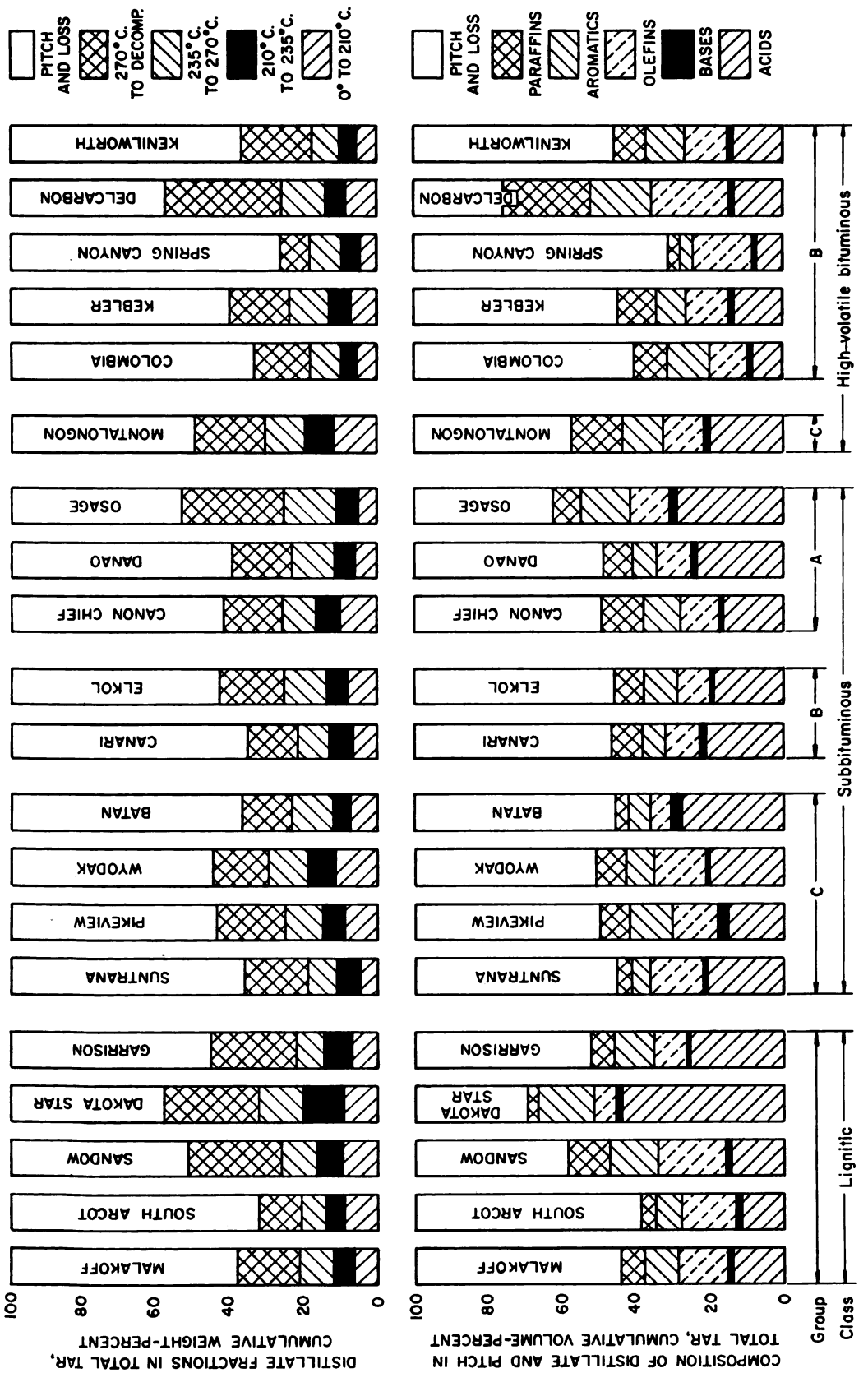

FIGURE 13. - Composition of Low-Temperature Tars From 20 Coals.

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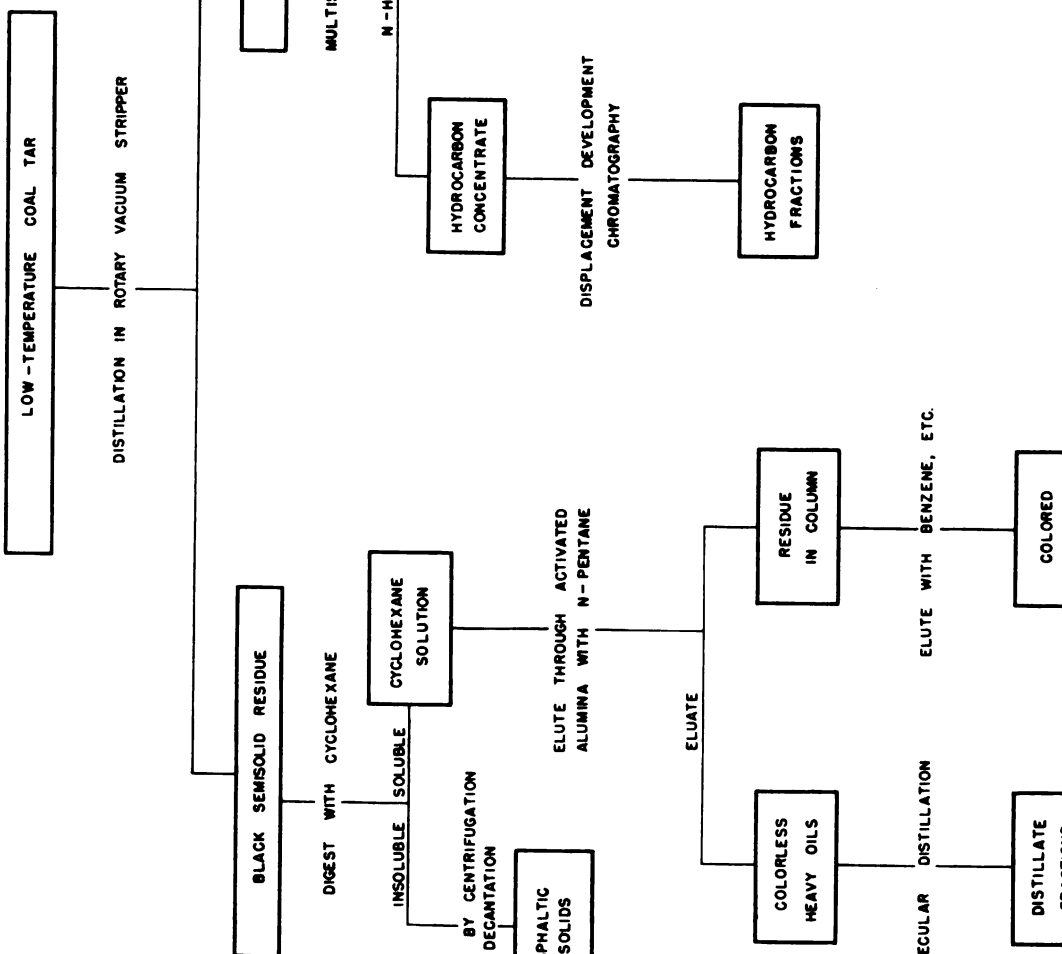

FIGURE 14. - Scheme for Separating Constituents of Low-Temperature Tar.

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Humic Acids

This general class of organic oxyacids is produced by both natural and artificial oxidation of all ranks of coal. Naturally oxidized lignite is available readily, and the alkali-soluble fraction (up to 80 percent) is being examined, partly as a potential source of pure carbon from subsequent thermal treatment. Humic acids obtained by alkaline extraction may be processed to an ash content as low as 0.22 percent by leaching with mineral acid. They may then be carbonized to produce tar and char analogous to that obtained from lignite.

Tar From Bituminous Coal

Separation and Characterization

Low-temperature tar from bituminous coal is being separated into its components to identify the chemical types present. Separation and characterization of tar components is necessary to determine the potential value of low-temperature tar for industrial nonfuel utilization.

A general separation scheme for low-temperature tar is shown in figure 14. The tar is distilled in a rotary vacuum stripper at temperatures below about 125° C. to avoid thermal alterations. A mixture of phenols is isolated from this distillate by multistage countercurrent extraction with n-hexane and a water-methanol mixture as the two essentially immiscible solvents. Phenols are recovered from the aqueous solvent, fractionated in a spinning-band vacuum still, and further split in a countercurrent distribution instrument shown in figure 15. This device employs 2 immiscible solvents in 60 cells or individual separation stages. The fractions from the spinning-band vacuum still are fractionated additionally in a gas-liquid-partition chromatography apparatus, as will be discussed further.

A series of runs was made with the rotary vacuum stripper to obtain tar distillate under very mild conditions. This distillate was treated further in the multistage, countercurrent extraction column to obtain phenolic and neutral oil concentrates. With the 11-stage column, the best possible results are a neutral oil and a tar acid extract, both of 97 volume-percent purity.

A sample of low-boiling tar distillate, representing that portion of the tar distillable at about 215° C., was extracted with Claisen alkali to give a phenolic mixture. A portion of this extract was fractionated in the spinning-band vacuum still to give seven fractions, each containing two to four individual phenols, readily identified by their infrared spectra.

A low-temperature tar pitch was partly characterized. The cyclohexane solubles were fractionated by elution chromatography with a column of activated alumina by means of a series of eluants with gradually increasing eluting power, which yielded a series of fractions varying from colorless oils to black semisolids. From refractive indices and molecular weights, approximations to the average structure were made.

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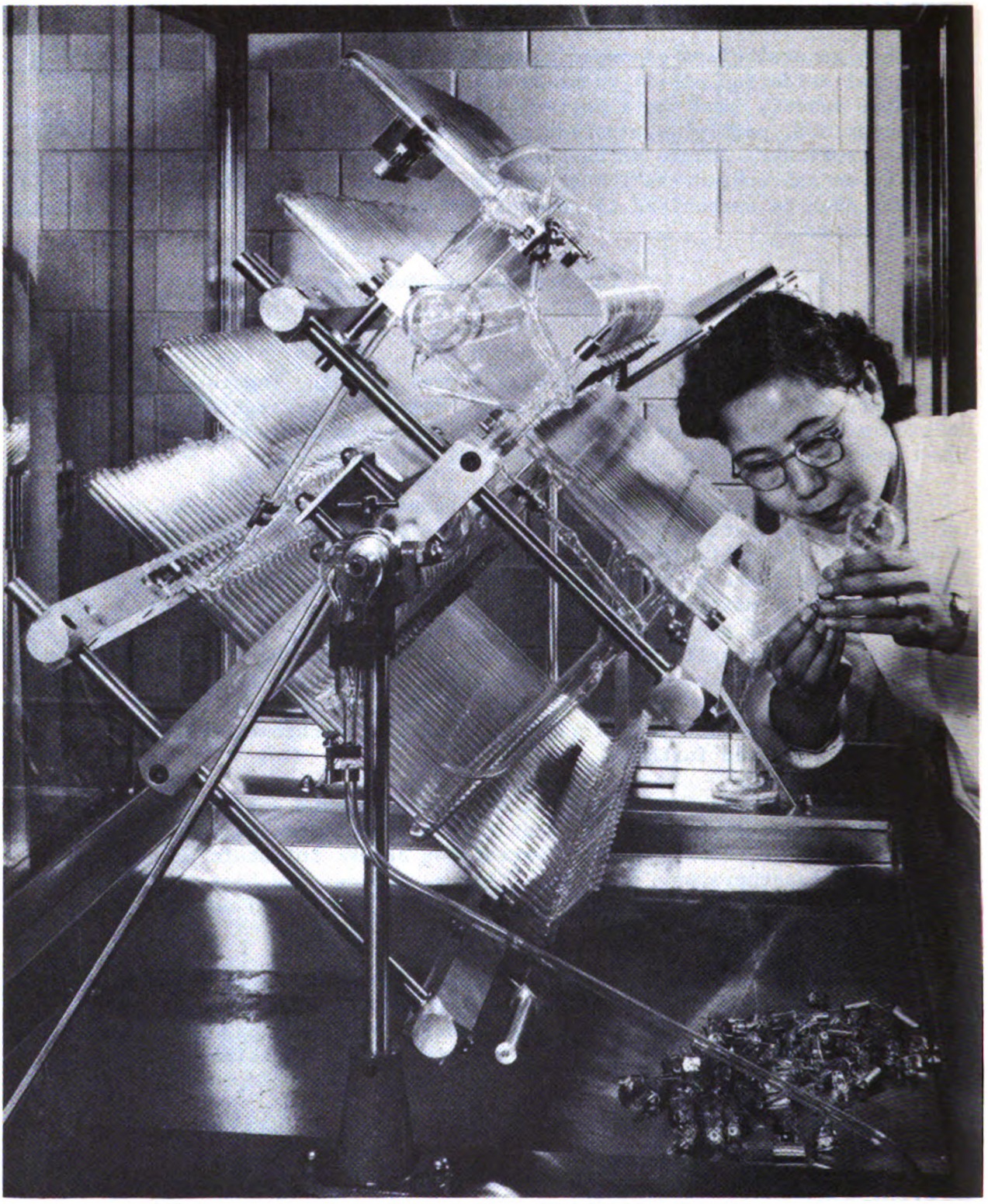

FIGURE 15. - Countercurrent Distribution Instrument for Fractionating Phenolic Compounds in Coal Tars.

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Upgrading

Owing to distinctive differences between low-temperature tar and coke-oven tar, refining processes and end uses established for the latter can seldom be applied to the former without modifications. Also, new techniques for fully utilizing the unique components of low-temperature tar must be developed.

One economically important characteristic of low-temperature tar is its high content of tar acids, especially high-boiling tar acids. A new separation process is being developed consisting of extraction with monoethanolamine, displacement by carbon dioxide, and regeneration of spent amine.

GASIFICATION OF COAL

Some of the unsolved problems of gasifying coal with steam and oxygen were reviewed.88/

Reaction of Carbon With Carbon Dioxide

The kinetics of the reaction of carbon with carbon dioxide has been investigated in the temperature range 700°-1,400° C. at atmospheric pressure, and a mechanism for the reaction was proposed.89/ The reaction involves transfer of an oxygen atom from carbon dioxide to a carbon atom in the solid and subsequent evaporation of the carbon-oxygen complex. Carbon monoxide molecules also remove oxygen from the solid, which retards the rate of oxidation of carbon and leads to a dynamic exchange of oxygen between monoxide and dioxide molecules. This study demonstrated that the reaction of carbon dioxide with carbons of different origin, particle size, porosity, and crystallinity can be treated on a common basis.

Reaction of Carbon With Steam

To measure the kinetics of the reaction of carbon with steam a tubular reactor was built in which the specimen is suspended in the center by a platinum wire attached to one arm of a balance while superheated steam is being passed through the tube. Reaction rates, measured by weight loss, are determined by removing small weights and measuring the time required for the balance to reach equilibrium.90/ Preliminary tests showed that the initial rapid rate of devolatilization was followed by a slower rate of reaction that remained constant up to 80 or 90 percent burnoff. The samples as a rule, were, allowed to react to complete burnoff; the ash residue had the same size and shape as the original sample.

88/ Newman, L. L., McGee, J. P., Oxygen Gasification of Coal. Some Unsolved Processing Problems: Ind. Eng. Chem., vol. 48, No. 7, July 1956, pp. 1112-1117.

89/ Ergun, Sabri, Kinetics of the Reaction of Carbon Dioxide With Carbon: Jour. Phys. Chem., vol. 60, No. 4, 1956, pp. 480-485.

90/ James, V. E., Study of the Effect of Carbon Particle-Size Ranges on the Steam-Carbon Reaction: M.S. Thesis, Univ. West Va., 1955.

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Flashing of Coal-Water Slurries

Investigation had shown that the rate of flow through a heated coil is the most important single variable causing particles of coal to break up when a coal-water slurry is flashed.91/ To verify this conclusion and to compare the behavior of various coals an improved coil reactor was constructed.

Twenty-one runs, three to five on each of five different coals, were made by the procedure described earlier. The coals included a lignitic subbituminous coal, a subbituminous coal, a poorly coking and a strongly coking bituminous coal, and a Pennsylvania anthracite. These runs confirmed the previous conclusions. As no significant differences were found among the five coals, rank and grade apparently had no appreciable effect on micronization.

Hydrogen by Steam-Iron Process

Pilot Plant

Although hydrogen for synthetic fuels may be made from coal by the reaction of coal, oxygen, and steam, savings in cost might be accomplished by avoiding the use of oxygen. An alternate method used industrially to make hydrogen on a small scale is the steam-iron process. Iron oxides may be reduced to metallic iron by producer gas and then oxidized with steam to produce hydrogen. These reactions have been obtained cyclically in fixed beds. The chief objections to their use on a large scale have been slowness of reaction and relatively large investment costs. An advantage of using producer gas as a reducing medium for the steam-iron process is that it is the cheapest gas that can be made from coal.

A small pilot plant has been constructed to determine the extent to which the rates of the reactions can be increased by using small iron particles (20- to 100-mesh) in a fluidized bed. In the reduction cycle producer gas is passed upward through the bed at velocities that will fluidize the solids. When the oxides of iron have been partly reduced to metallic iron, steam is admitted to the bottom of the reactor to convert the iron to oxide and produce hydrogen. The vessel in which the reaction is studied is a 2-inch-diameter tube of Schedule 160, Type 316 stainless steel, long enough to accommodate a 5-foot fluidized bed. Solids are fed to the top of the bed through a charging hopper and discharged from the bottom of the bed through a receiving hopper. Thus far, the unit has served only to learn techniques of fluidizing and handling solids at the high temperature of the reaction.

Solids-Transport Studies

In the steam-iron process solids from a fluidized bed at high temperatures must be circulated between a reduction unit and an oxidation unit. The power requirements are being investigated for transporting solids from one vessel to another by means of compressed gases. Because of anticipated

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91/ Salgado, P. G., The Effects of Variables in Flashing Coal-Water Slurries in Heated Coils: M.S. Thesis, Univ. West Va., 1955.

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mechanical difficulties with lock-hopper valves at pressures up to 450 p.s.i.g. and temperatures up to 900° C. a solids-seal leg is being considered for moving hot solids from the reduction vessel into the oxidation vessel.

A bench-scale standpipe system has been constructed to study factors affecting the transport of solids, such as pressure gradient and gas leakage at various flows of 60- to 200-mesh magnetite. The original test section was constructed of 2-inch Pyrex pipe; 4-inch steel pipe currently is being used. The values of the four main variables, flow of solids, bulk density, gas rate, and pressure drop were determined. The maximum pressure gradient in the 2-inch pipe was 1.005 p.s.i. per foot of bed height. This peak occurred at a flow of 20 pounds of solids per minute, equivalent to about 36,000 pounds of solids per hour per square foot of cross section.

Gasification of Lump Coal

Gasification of coal with an oxygen-steam mixture for synthesis or heating purposes is becoming increasingly important in the future overall national energy picture. Due to relatively low oxygen requirements and high carbon conversion, gasification of lump fuel in a fixed bed with oxygen and steam at elevated pressure is particularly attractive as far as economy of gasification is concerned. This type of processing is especially suitable for low-rank fuels, like lignite and subbituminous coal, because of their high reactivity and nonagglomerating characteristics. Data are being obtained on process variables and equipment, on the operability of a gasifier, and on optimum conditions for gasification of lump lignite with oxygen and steam at elevated pressure.

During the past year work proceeded on two major phases: (1) Design of a gasification pilot plant, and (2) study of the thermodynamics of pressure gasification and design of statistically planned experiments.

Consideration was given to the design of a slagging gas generator; modifications were included to permit conversion to "dry bottom" or nonslagging operation. A contract was let for a preliminary engineering report and a cost estimate for the proposed pilot plant. These reports have been received, and some equipment has been purchased. A scale model of the plant has been fabricated to facilitate layout and for preliminary training of operators.

Work was begun on thermodynamic calculations to provide a basis for estimating material requirements for pressure gasification as well as yields, composition and temperatures of gas. Calculations completed to date on char indicate that a high equilibrium temperature is favorable for producing mixtures of H_2 + CO. Oxygen-steam molar ratios between 0.7 and 1.0 appear to be best suited for preliminary experiments.

A series of statistical experimental designs was formulated for possible use in the gasification program. Four possible basic designs were selected to determine flexibility and operability of the gasifier.

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Data on cost of construction and operation of an externally heated gasifier were published.92/ For a plant producing 50 million cubic feet (H_2 + CO) per day, the estimated operating cost for gasifying as-received lignite was 37.6 cents per thousand cubic feet, and for gasifying steam-dried lignite, 32.0 cents.

A paper on the technology and utilization of lignite presented information on reserves, mining techniques, and composition of lignite.93/ Methods and equipment for industrial combustion as well as for nonfuel uses were discussed. Reference was made to present studies on carbonization and gasification.

Gasification of Powdered Coal

Pilot-Plant Operation

During the current year the high-pressure pilot plant for gasifying pulverized coal with steam and oxygen was operated for over 600 hours in more than 100 tests. Pressures were varied from 150 to 300 p.s.i.g., and feed rates from 200 to 400 pounds of coal per hour per cubic foot of reaction volume. The silicon carbide refractory lining was in good condition after 270 hours of operation. It was then renewed because of modifications to other parts of the gasifier and was in good condition after about 300 hours of operation.

All tests were made with bituminous A coal from the Sewickley bed in Monongalia County, W. Va. The work followed a factorial design, which permitted statistical analysis of the data. It was designed to determine the effects of coal throughput, pressure, steam-oxygen-coal ratios, and heat loss on the capacity of the gasifier and the efficiency of carbon utilization. Heat loss is an important factor; however, because it depends on the construction of the reactor (size, type of refractory lining, amount of cooling needed to protect equipment) as well as on other variables, it cannot be held constant for each test condition. Consequently, three series of experiments were planned to vary the heat loss, as determined by construction of the gasifier. The first series was made with the entire reaction volume water cooled. In the second series heat loss was about one-half that of the first. For the third series of tests the lining will be modified to give a heat loss about 25 percent less than that with present construction.

The coal-preparation facilities were improved, so that larger and more uniform batches of coal are available for gasification. In a 16-hour test under steady conditions hourly variations in analysis of coal and degree of pulverization were small.

The percentage of carbon gasified in the first series of tests (water-cooled wall) was varied by changing the rate of coal input, the pressure, and

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92/ Ongstad, O. C., Chetrick, M. H., and Oppelt, W. H., Cost Data for Gasification of Lignite in an Externally-Heated Retort: Bureau of Mines Rept. of Investigations 5272, 1956, 17 pp.

93/ McMurtrie, R., and Oppelt, W. H., Developments in Lignite Technology and Utilization: Ind. Eng. Chem., vol. 48, No. 11, 1956, pp. 1996-2000.

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the ratio of oxygen to coal fed to the gasifier. The steam-coal mass ratio was constant at 0.3. An equation used to correlate the data accounted for 93.5 percent of the variation; 6.5 percent was left for random error. The standard deviation of a single observation was 2.6 percent of carbon gasified.

At the lowest pressure gasification decreased as the coal rate increased, indicating that a decrease in residence time (the time spent in the hot zone of the gasifier) causes a decrease in carbon gasification. At the highest pressure, on the other hand, the percentage of carbon gasified increased as the coal rate increased, indicating that the capacity of the gasifier had not been reached. This result may be attributed to loss of heat, because heat loss per unit of coal decreased as throughput increased.

Coal-Feeding Methods

In continuing development of a method of feeding a pressure gasifier with coal and superheated steam derived from a coal-water slurry, emphasis has been placed on the design of a suitable pipe coil for heating the slurry. To avoid problems associated with swelling and coking during pyrolysis nearly all development work was done with an anthracite-water slurry. All abrupt changes in direction have been eliminated, because they caused erosion of pipe walls. A composite coil, consisting successively of 1/2-, 3/4-, and 1-inch pipes, was most effective. A more uniform velocity was obtained consistent with changes in the mixture as it passed through the coil.

Nuclear Energy for Coal Gasification

Cooperative work is continuing with AEC on the use of nuclear fuels to supply heat for the steam-carbon reaction.94/ The following phases of the work are being studied by the Bureau: (1) Testing simulated-fuel elements in various atmospheres by both electric induction and resistance heating; (2) helping procure compressors to recycle gas at 1,000° F. and above; (3) removing impurities from recycled gas at elevated temperatures and pressures; (4) making economic evaluations of various uses for nuclear-process heat; (5) calculating heat transfer, pressure drop, and energy requirements for various reactor geometries.

Four schemes now are under consideration for the application of nuclear heat to coal gasification:

Scheme 1. - Direct injection of steam and coal into a nuclear reactor offers the advantage of having complete reaction take place in one vessel; however, this method puts the maximum burden on the reactor. The fuel elements will be subjected to slag erosion, which complicates the already formidable problem of fuel-element cladding. Irradiation tests on anthracite, bituminous and subbituminous coal, and coal ash showed that most of the radiation was

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94/ Strimbeck, G. R., McGee, J. P., Katell, Sidney, Atomic Energy in the Gas Industry: Am. Gas Assoc. Monthly, vol. 38, No. 10, October 1956, pp. 35-36, 50-52.

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absorbed by the ash, making the disposal problem almost insurmountable. For this reason the direct-injection method is least promising.

Scheme 2. - Using recycled gas as a heat carrier means passing a comparatively clean stream through the reactor. The German Wintershall-Schmalfeldt gasification process made use of recycled synthesis gas. The top temperature was low (2,250° F.), and a low-rank reactive coal was used. Nuclear heating of synthesis gas would offer the advantage of a continuous process and allow gasification at 30 atmospheres. Fuel elements would have to be clad with a material impervious to the gas and also to most of the fission products. Separating dust and ash from the recycle stream appears to be a difficult problem. A waste-heat boiler to cool the recycle gas to 1,400° to 1,500° F. would allow gross separation by a concentrator cyclone. Since steam is required for the process, a Venturi scrubber, operating at saturation temperature with recycled scrubbing water, should be investigated. The steam and heat balance will determine the feasibility of such a scrubbing system. A compressor for recycling synthesis gas at 1,400° F. would have to be developed; however, a recirculating compressor is available for use at 1,200° F. and 250 p.s.i.g.

Scheme 3. - The nuclear reactor could be used to preheat steam, which would eliminate the dust problem and the need for a recycle compressor. However, owing to loss of latent heat of steam, which cannot be recovered in a waste-heat boiler, steam would have to be heated at least to 3,000° F., and preferably 3,500° F., so as to keep steam requirements reasonable. Cladding for fuel element to operate at these temperatures is the major problem. Adding oxygen, preheated in the reactor, would allow lower steam temperatures. This system is attractive because it is a modification of the present gasification technique rather than an entirely new approach. For example, previous work by the Bureau has shown that with steam preheated to 3,500° F. oxygen requirements are reduced by two-thirds.

Scheme 4. - Recycling inert gas as the heat carrier and indirect heat exchange to the coal-steam mixture is the approach favored by nuclear engineers to take advantage of using a nuclear reactor containing cheaper fuel elements and to permit continuous removal of fission products; however, this system transfers many problems from the reactor to the gasifier-heat exchanger. For gasifying lignite and possibly some subbituminous coals, a metallic heat exchanger might be used. It would be expensive, as alloy steels would be required. Both helium and process streams could be at 30 atmospheres to aid transfer of heat. Also, the gas could be generated more economically at 30 atmospheres instead of 1 atmosphere for subsequent use at that pressure. This system could be adapted easily to reforming off-gases for producing hydrogen. For bituminous coals, a ceramic heat exchanger would be required. One has been built, although several companies are willing to build one.

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Study of Materials and Problems Related to Use of Nuclear Energy

One approach to simulating conditions in a gas-cooled nuclear reactor has been to develop procedures for testing the properties and reactions of materials under the severe conditions of nuclear heating. The first simulated nuclear reactor was described in the previous report. Several attempts to gasify powdered coal suspended in steam by passing this suspension through the reactor ended in failure of the silicon carbide at 2,500° to 2,800° F.

Helium cycling was tested for 1 month at 2,500° F. with five 1-1/4-inch-diameter "KT" silicon carbide balls in the reactor. For 3 weeks the helium coolant was heated in the reactor to above 2,250° F. and continuously recycled at a rate of 1.25 c.f.m. During the fourth week the rate of flow was varied from 0.50 to 3.0 c.f.m. for heat-transfer measurements. Although commercial grades of silicon carbide balls had failed mainly because of decomposition of the bonding material, this test demonstrated the feasibility of using "KT" silicon carbide (binder free, but containing a slight excess of free silicon) as construction material.

For further testing the original reactor was reconstructed. The second reactor (see fig. 16) was designed so that the tube extended beyond the shell to get better seals on top and bottom. It also was heated electrically, but a carbon cylinder was used as the conductor in place of the structurally weaker spiral carbon element. Slow disintegration of the carbon, due to reaction with traces of moisture in the insulation, nevertheless continued to cause difficulties and failures in heating. For this reason, a third reactor was designed in which the Globar reactor tube itself will be used as an electric resistor. This modified reactor is now ready for testing.

Among the various ceramic and cermet materials evaluated, the following deserve mentioning:

Silicon carbide. - The durability of SiC depends chiefly on the binding material. Silicon nitride-bonded SiC was reduced slowly at 2,500° F. and more rapidly at 2,800° F.; as a result the bonding agent was removed, and the sample disintegrated. "KT" silicon carbide withstood temperatures up to 3,000° F. in a neutral or reducing atmosphere; in oxidizing atmospheres a protective coating of silica formed, which extended the life of the material.

Nickel aluminide. - This alloy (m.p. 3,000° F.) was inert to reducing or neutral atmospheres at 2,500° F. but slowly reacted with steam or other oxidizing atmospheres (1 percent conversion per hour).

Molybdenum disilicide. - This material was inert to all atmospheres at 2,500° F. It had poor thermal shock resistance compared with the preceding two materials yet considerably better than most ceramic materials.

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Zirconia. - Newer grades of stabilized ZrO_2 (CaO used as stabilizing additive) had fairly good thermal shock resistance and were completely inert to gaseous reactions at 2,500° F.

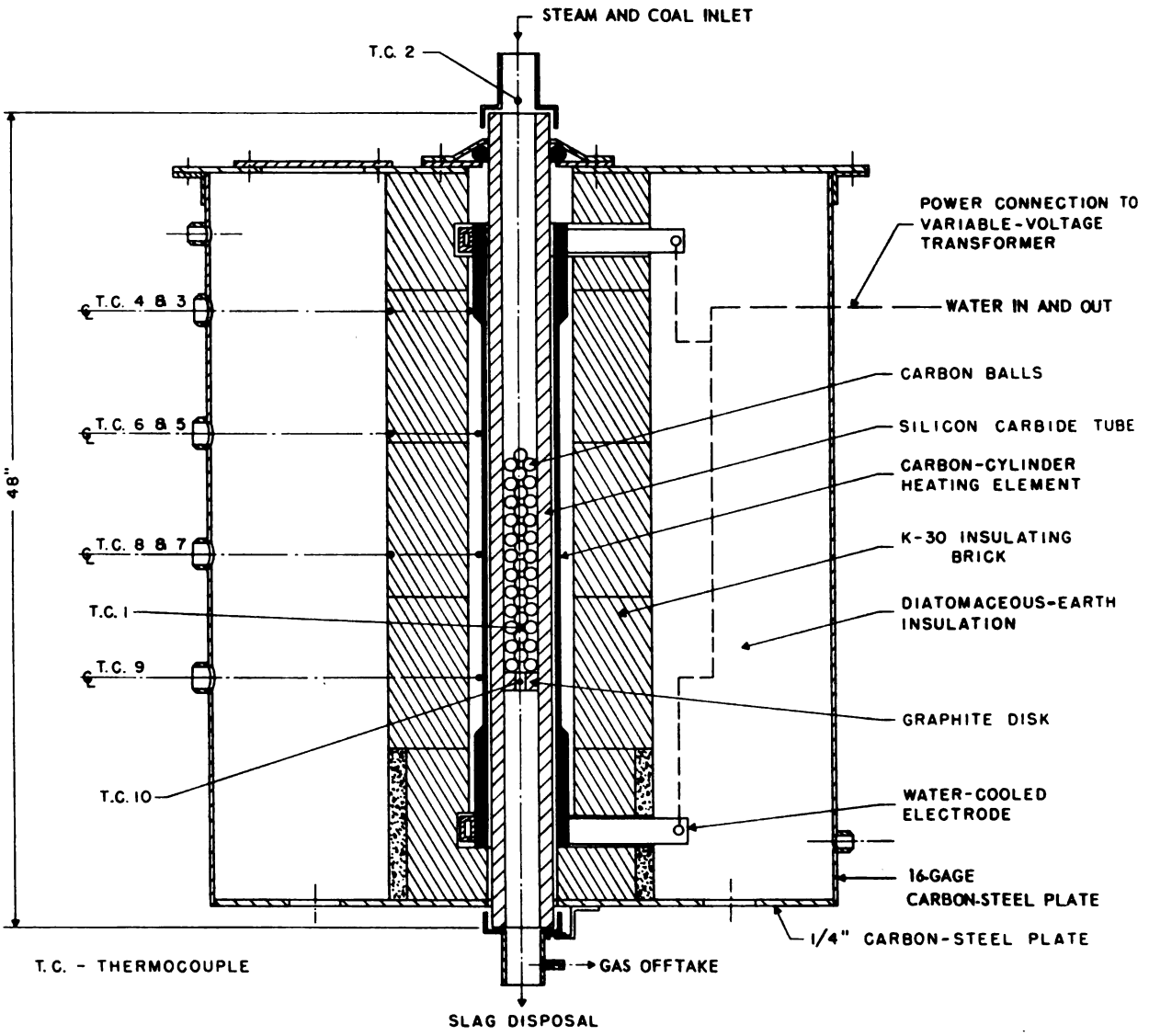

FIGURE 16. - Resistance-Heated Simulated Nuclear Reactor (RBR-2).

Induction-Heated Reactor

When an apparently suitable material has been found as the result of the small-scale tests described above, it must be tested further for longer periods and on a larger scale. For this purpose an induction-heated reactor has been built. The reactor consists of a stainless-steel shell, refractory lined, with a refractory tube of silicon carbide encased by a water-cooled

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induction coil. The power unit is a 100-kw., 10,000-c., 400- to 800-v. motor generator. The induction load consists of coated graphite balls. Tests are underway now on various unclad graphite shapes.

A suitable inductor, two parallel five-turn coils, was assembled in the reactor. Inert gas was passed through the reactor at constant ball temperatures of 1,500° and 2,000° F., while exit-gas temperatures and energy distributions were measured. When the gas-recycle system was redesigned so that the gas could be preheated to 1,000° F. before entering the reactor, exit-gas temperatures of 2,500° F. were obtained easily.

Since interest currently centers on materials to be used in a helium atmosphere, a helium-recycle system was designed with an electrical preheater in which the gas will be heated to approximately 1,000° F. before passing through the reactor. After the helium is heated in the reactor to 2,500° F., it will pass through a hot dust-filter box to remove any dust picked up from the cladding and the tubes. This box is designed for minimum pressure drop through the high-temperature filter.

Gas Purification

Raw synthesis gas made from coal contains several impurities detrimental in subsequent processing - especially sulfur (both organic and inorganic), solids, and carbon dioxide. Their removal is discussed in a recent paper;95/ another is concerned with the purification equipment at the former demonstration plant of the Bureau.96/

Purification by Resins

Several resins were evaluated for removing carbonyl sulfide, hydrogen sulfide, and carbon dioxide by ion exchange from mixtures of gases. Amberlite IRA-400 and Amberlite IRA-410 resins were tested in both the hydroxyl and chloride form. The chloride form of the resin removed only slight amounts of hydrogen sulfide or carbonyl sulfide, but the hydroxyl form removed about 8.4 grams of sulfur per 100 grams of resin when tested with a gaseous mixture containing hydrogen sulfide. Carbonyl sulfide also was removed fairly well with the hydroxyl form, but little absorption of carbon dioxide was noted.

Purification by Diethanolamine

Tests on an agitated absorber have shown that diethanolamine solution removes both hydrogen sulfide and carbon dioxide to below the upper limits for synthesis gas when the liquid-gas ratio is high. Sequential factorial experiments were made to determine the effects of several factors and their interactions under different conditions.

95/ Wainwright, H. W., Kane, L. J., Wilson, M. W., Shale, C. C., and Ratway, J., Purification of Synthesis Gas: Removal of Dust Carbon Dioxide, and Sulfur Compounds: Ind. Eng. Chem., vol. 48, No. 7, July 1956, pp. 1123-1133.

96/ Willmott, L. F., Batchelder, H. R., Wenzell, L. P., Jr., and Hirst, L. L. Performance of a Girbotol Purification Plant at Louisiana, Mo.: Bureau of Mines Rept. of Investigations 5196, 1956, 18 pp.

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Purification by Hot Potassium Carbonate Solutions

Development was continued of a scrubbing process operating at elevated pressure and employing a hot solution of potassium carbonate as absorbent. Because absorption occurs at about the same temperature as regeneration of the spent solution, no heat exchange is necessary between these two steps, and less steam is required for regeneration than in conventional processes. The decreased consumption of steam lowers the cost of purification.

Bench-scale studies were made to determine the equilibrium pressure of hydrogen sulfide over solutions of potassium carbonate at temperatures and concentrations of interest for plant operations. Solutions containing 20 and 30 percent potassium carbonate were investigated at selected carbonate-bicarbonate ratios and concentrations of hydrogen sulfide. Equilibrium pressures of hydrogen sulfide, carbon dioxide, and water vapor above these solutions were measured at 70° to 130° C. These data are required for determining the efficiency of purification and evaluating pilot-plant results.

Impure gases containing 10 to 20 percent carbon dioxide and 0.2 to 1.0 percent hydrogen sulfide were used in pilot-plant tests; the range of concentrations resulted from gasification of coals of low- and high-sulfur content.[97] About the same fraction of the entering carbon dioxide and hydrogen sulfide were removed by hot carbonate scrubbing, and partial pressures of hydrogen sulfide in the purified gas closely approached equilibrium values. With a feed gas containing 11 percent carbon dioxide and 0.5 hydrogen sulfide, the residual concentration of hydrogen sulfide was about 0.03 percent when the carbon dioxide concentration was lowered to 2 percent and 0.004 percent when the carbon dioxide concentration was reduced to 0.30 percent. Carbonyl sulfide was completely removed from feed gases containing 10 to 50 grains of this component per 100 cu. ft. Consumption of steam for regeneration was about the same as when carbon dioxide alone was removed.

Dust Removal

Dust is removed from synthesis gas in the pilot plant in two water scrubbers in series, an unpacked tower scrubber and a Venturi scrubber. No solution yet has been found to the problem of sampling a saturated gas under pressure to determine its dust content, so the concentration of dust at the inlet to the tower scrubber is unknown. A bench-scale scrubber is under investigation to determine its efficiency under the same conditions that exist in the plant. Work at atmospheric pressure and temperature is underway. A pulsating dust feeder was developed for dispersing known quantities of dust of a given size range into a gas at atmospheric pressure to serve as feed to the small scrubber.

Gasification of coal by nuclear energy raises new dust problems. Almost complete removal at high temperatures and pressures is necessary because of the potential radioactivity of the dust - in addition to purity requirements.

97/ Benson, H. E., Field, J. H., and Haynes, W. P., Improved Process for CO_2 Absorption Uses Hot Carbonate Solutions: Chem. Eng. Prog., vol. 52, No. 10, October 1956, pp. 433-438.

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The most satisfactory commercial material found thus far for high-temperature operation is an aluminum silicate fiber. Many initial problems of filtering gas at elevated temperatures have been solved, but the maximum operating temperature in small apparatus to date has been 1,800° F.

Analysis for Naphthalene

Under certain conditions gas made from coal contains naphthalene. A rapid method was developed for its quantitative analysis in gases. A known quantity of gas is bubbled through a measured volume of picric acid solution of known concentration; any naphthalene present reacts with picric acid to form a colloidal precipitate of naphthalene picrate, which is measured by a spectrophotometer. This method is more accurate, quicker, and safer than conventional methods.

Cost of Air Compression for Oxygen Plants

Interest in tonnage oxygen plants led to a study of air-compression costs - the major portion of the operating cost. Capital costs for the compressors were obtained from vendor quotations. The required investment costs for the unit (installation, foundation, piping, instrumentation, building, etc.) were evaluated. Numerous discussions with operators of air-compression units verified the fact that the major item of operating cost is power. The cost of operating a 20,000-c.f.m. unit at 50 p.s.i.g. increases 60 percent with an increase in power costs from $0.0075 to $0.015 per kw.-hr. For a 60,000-c.f.m. unit at 100 p.s.i.g. this increase amounts to 88 percent for the same change in power costs. With power at $0.01 per kw.-hr., the operating costs were calculated to range from $0.031 to $0.044 per 1,000 cu. ft. for a 20,000-c.f.m. unit operating at discharge pressures of 50 and 120 p.s.i.g., respectively. These costs drop to $0.024 and $0.036 for a 60,000-c.f.m. unit.

Underground Gasification

Field scale experiments are being conducted on underground gasification to obtain chemicals and/or energy from coal in situ. The primary objectives include utilization of coalbeds uneconomical for mining and of coal remaining in areas where mining has been completed. The work is being done under a cooperative agreement between the Bureau of Mines and the Alabama Power Co., at Gorgas, Ala., in the America coalbed, which is double-bedded, heavily cleated, and about 5 feet thick, including 10 to 11 inches of fire-clay parting.

Gasifying coal underground requires a passage in the coalbed connected to the surface by inlets and outlets. Gas-making fluids (air, oxygen, or steam) are injected into the underground reaction zone after ignition is accomplished, and the products are obtained at the outlet.

Hydraulic Fracturing

During 1956 investigations were concerned with preparing the passage by application of hydraulic pressure to fracture the coalbed. This passage is

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made accessible to the surface by boreholes. Gasification paths then are developed between the holes by a procedure known as "backward burning." Figure 17 shows the place of initial fracture, (HF-2) and the borehole system for testing the feasibility of backward burning.

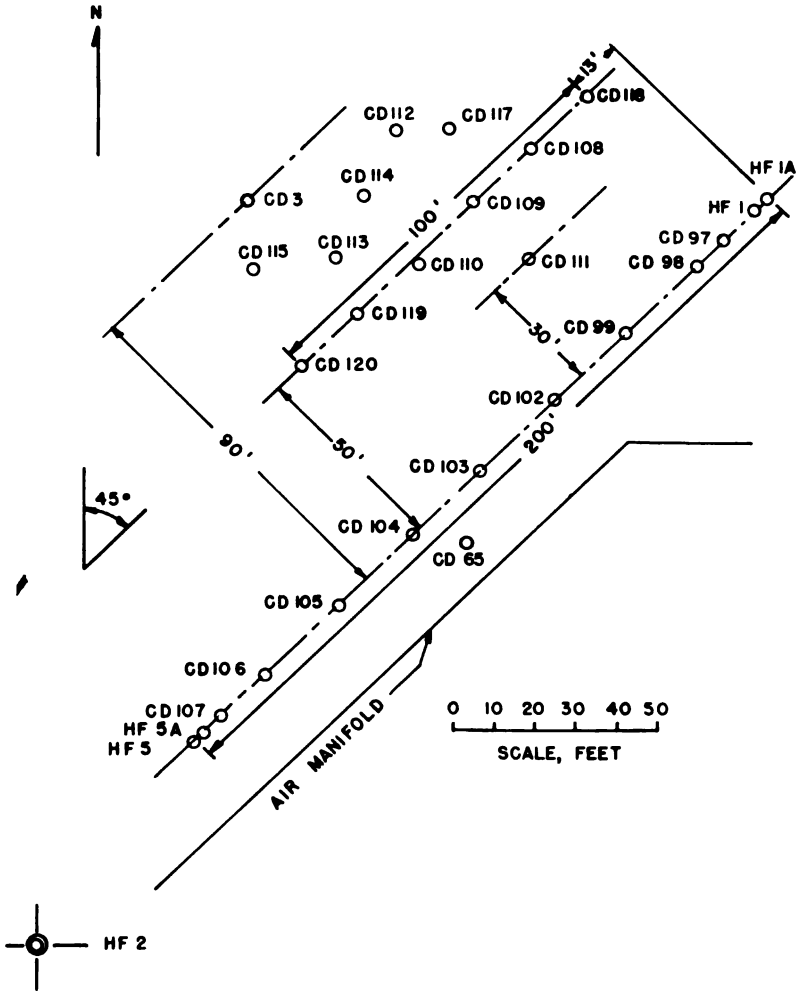

FIGURE 17. - Borehole Plan, First Hydraulic Fracture Experiment, Gorgas, Alabama.

Preignition permeability tests were made after hydraulic fracturing by injecting air at HF-1 and measuring airflow from the other wells. The gross air recovery was 62 percent at an initial injection pressure of 128 p.s.i. and a final pressure of 87 p.s.i. Eighty-five percent of the recovered air was obtained over the first 95 feet of path, and the remaining 15 percent was distributed along the last 100 feet.

Ignition was accomplished by a propane burner inserted at HF-1 into the coalbed. Preparation of a reaction path from HF-1 to HF-5 was begun immediately, with backward burning and the intermediate boreholes as steppingstones. The air-injection rate was 206 c.f.m. at a pressure of 102 p.s.i. An average of 26 percent of the injected air was recovered as product gas. Under the conditions obtained when preparing the path by backward burning, the theoretical amount of energy required for air compression was 75 percent of the energy available as heat of combustion of the product gas. In 74.4 days a gasification path was established having a nominal length of 200 feet; the average rate of preparation was 2.7 feet per day. Actual rates varied between 0.8 and 11.6 feet per day. Although the reasons for the different rates are unknown, one possibility is that the cross-sectional area of the path (or multiplicity of paths) has a greater effect on the rate than does length of the path (deviation from a straight line). The average cross sectional area for the whole system was 14.7 sq. ft. During path preparation the product gas had an average heating value of 62 B.t.u. per cubic foot.

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After final breakthrough, 3 days were used to improve those sections of the path that showed excessive pressure drop. Unidirectional gasification was then started with HF-1A as an inlet and HF-5 and HF-5A as a common outlet. The air-injection rate was increased throughout the operation. At times water was injected upstream of the outlet to control the outlet temperature. Gasification was maintained for about 30 days.

After gasification between HF-1 and HF-5 was stopped, injection wells CD-108 through CD-118 were drilled adjacent to the reaction zone. Path development and gasification operations were conducted in a manner similar to that between HF-1 and HF-5. Thus this method might be used to develop a whole area.

Electrolinking

Small-scale electrolinking equipment was installed, and 32 experiments were conducted on a nearby deposit of the Pratt coalbed. The overburden was removed to within 10 or 12 feet of the coal over an area of 60 by 100 feet. All but seven trials were made with horizontal electrodes inserted at various spacings and elevations on the face. In the last seven tests, holes were drilled from the surface, and vertical 4-inch-diameter electrodes placed in the coal. Horizontal electrode spacing ranged from 2-1/2 to 10 feet; the majority of the tests were made with a 3-foot spacing. The vertical electrode spacing was 5 feet in four trials, 7-1/2 feet in two trials, and 20 feet in the last trial. In 18 of these tests it was possible to establish a path by electrical linking between electrodes. No linkage was obtained when the electrode spacing was more than 7-1/2 feet. It was impossible to link the 20-foot vertical electrodes because of inadequate power. The tests indicated that electricity is conducted preferentially through fusain bands and partings in the coal.

COMPLETE CONVERSION OF COAL TO FLUID FUELS

Investigations were continued to improve processes for producing liquid and gaseous fuels from coal, and fundamental research was continued on the catalytic reactions involved. The long-range objective of this program is to provide scientific and engineering information that will be useful to American industry when synthetic fuels from coal will be required in the United States. The immediate objectives are to decrease the cost of synthetic fuels and improve the quality of products by developing new or improving known processes. The program on complete conversion of coal is divided about equally into studies of direct hydrogenation of coal and hydrogenation of carbon monoxide (Fischer-Tropsch and related processes).

In a discussion of a paper, Future of Synthetic Liquid and Gaseous Fuels, by H. R. Batchelder and H. W. Nelson (Mech. Eng., vol. 78, January 1956, pp. 11-14), Bureau research and development on synthetic gaseous and liquid fuels was cited as a vital factor in reducing the price differentials between the natural and synthetic products and bringing closer the time when commercial production of these fuels from coal will be economical.98/

98/ Corey, R. C., Synthetic Fuels: Mech. Eng., vol. 78, No. 7, July 1956, pp. 660-661.

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Coal Hydrogenation

Autoclave Studies

Investigations in batch autoclaves, the "test tubes" of coal hydrogenation, included studies of the nature of catalysis by iron compounds, of metal naphthenates as catalysts, and of the influence of large quantities of alkali hydroxides.

The reactions of metallic iron, ferric oxide, magnetite, ferrous sulfate and ferrous chloride with hydrogen, hydrogen sulfide, and mixtures of these two gases have been investigated at elevated temperatures and pressures in batch autoclaves to obtain some insight into the chemical state of iron catalysts during coal hydrogenation. Temperature, pressure of hydrogen, and pressure of hydrogen sulfide may have considerable influence on iron-catalyzed coal hydrogenation because of their effect on chemical transformations of iron compounds. The reactions of some iron compounds with hydrogen sulfide near atmospheric pressure were comparable in rate to reactions with hydrogen at high pressures. The order of decreasing reactivity to hydrogen and hydrogen sulfide was metallic iron and iron oxides, ferrous sulfate, and ferrous chloride. Temperatures of about 400° to 450° C. were required for the reaction of ferrous sulfate and about 500° C. for that of ferrous chloride.

If the reaction of a catalyst with hydrogen sulfide is an important step in the sequence of reactions involved in coal hydrogenation, the quantity of hydrogen sulfide available at a particular time may be a rate-determining factor, particularly at relatively low temperatures, such as 400° C. Appreciable amounts of hydrogen sulfide are produced between 350° and 400° C. from uncatalyzed Rock Springs, Wyo., coal containing about 1 percent sulfur. The amount of hydrogen sulfide formed at 400° C. would be enough to sulfide only about 20 percent of 1 gram of sulfur-free iron catalyst per 100 grams of coal.

A study is progressing on the effect of hydrogen sulfide in the iron-catalyzed hydrogenation of coal and of asphaltene, a high-molecular-weight product of coal hydrogenation.

The walls of a new, type 347, stainless-steel autoclave were not catalytically active in hydrogenating coal at about 450° C. and 8,000 p.s.i. Treating the autoclave with hydrogen and hydrogen sulfide did not cause the walls to become active, although visual observation indicated that hydrogen sulfide had reacted with the stainless steel. Memory effects, due to small amounts of catalysts retained in the system from previous experiments, are being investigated for tin catalysts.

The catalytic activity of naphthenates of molybdenum, nickel, tin, iron, cobalt, chromium, zinc, and copper in concentrations of 0.1 percent metal, based upon coal, was investigated in a rotating autoclave. The experiments were made under 3,000 p.s.i. initial hydrogen pressure and at "zero time," that is, the charge was heated to 500° C. then slowly cooled. Rock Springs coal was used alone and with middle oil as the vehicle. With dry coal the naphthenates of molybdenum and nickel gave the highest conversions (97 percent), but only molybdenum produced oils very low in asphaltene. Tin, iron,

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cobalt, zinc, and copper naphthenates impregnated on coal gave conversion of 82 to 90 percent. Chromium naphthenate on dry coal was ineffective. Hydrogenation in the presence of vehicle oil produced essentially the same results. Asphaltene yields were lowest, and nitrogen elimination highest with molybdenum. The mode of adding the catalyst appears unimportant when naphthenate catalysts are used for hydrogenating coal in the presence of vehicle oil.

The effect of sodium hydroxide on the hydrogenation of coal in the presence of stannous chloride was studied at 500° and 525° C. for 15 minutes at temperature with an initial hydrogen pressure at room temperature of 2,000 p.s.i. The amount of sodium hydroxide used ranged from 2.7 to 107 percent of the weight of the coal. Within these limits, the concentration of sodium hydroxide had no effect on conversion, which was about complete in all tests, or on the yields of heavy and light oil. As the amount of sodium hydroxide was increased, the yield of asphaltene decreased and the yield of carbon dioxide (as sodium carbonate) increased. The consumption of hydrogen decreased with increasing concentrations of alkali.

Analysis of Phenols

The analysis of Fischer-Tropsch alcohols by conversion to their trimethylsilyl ethers and subsequent mass spectrometric analysis has furnished a rapid and accurate method of characterizing synthesis products. The method now has been extended to the characterization of tar acids from coal hydrogenation, and the results appear promising for analyzing complex mixtures of phenols. Trimethylsilyl ethers of phenol, the three isomeric cresols, α-naphthol, resorcinol (1,3-dihydroxybenzene), and 2,6-di-tert-butyl phenol have been synthesized and their mass spectra determined.

A paper was published on correlating the ultraviolet spectra of a number of aromatic compounds, most of which occur in coal-hydrogenation products.99/

Pilot-Plant Operation

The demands for safety and operability of equipment for coal hydrogenation have required a variety of new or modified experimental methods.100/

Conventional Hydrogenation of Coal

Extensive engineering studies have been made on the continuous hydrogenation of coal at 1,500 to 3,500 p.s.i.g. and 420° to 480° C. These experiments dealt primarily with the effect of residence time, pressure, temperature, and type and concentration of catalyst on the production of high-boiling fuels. During the last few years an attempt has been made to produce gasoline in a single-step operation at higher temperatures (500° to 525° C.), large concentrations of an active catalyst (about 1 percent), and partial pressures

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99/ Friedel, R. A., Correlations in Ultraviolet Spectrophotometry: Appl. Spectroscopy, vol. 2, No. 1, November 1956, pp. 13-24.

100/ Hiteshue, R. W., Bench and Pilot-Plant Practice: Ind. Eng. Chem., vol. 48, No. 5, 1956, pp. 835-837.

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of hydrogen (8,000 p.s.i.g.). This objective was never achieved because reactants "coked" at these high temperatures in spite of carefully controlled operations. Temperature control under these extreme conditions was possible only through the use of small concentrations of catalyst. Much of 1956 was devoted to a study of the conventional process at 8,000 p.s.i.g. and 460° C. with very low concentrations of an active catalyst for producing distillable oils. Before this, the minimum concentration of catalyst required for satisfactory operation had been believed to be 0.5 to 1.0 percent - quantities that cannot be used commercially owing to economic reasons. Recent research showed that a decrease in concentration of molybdenum from 1.0 to 0.01 percent resulted in little or no loss in conversion and only a moderate increase in the production of asphaltene. Thus, the purpose of these studies was to determine the feasibility of using 0.01 percent molybdenum and to determine the effect of residence time on distribution of the product in an equilibrium recycle operation in which no extraneous oils are added to the system. Rock Springs, Wyo., coal, impregnated with 0.01 percent molybdenum as ammonium molybdate, was hydrogenated at 460° and 480° C., 8,000 p.s.i.g., and coal-paste rates of 15 to 50 lb. per hr. Coal, with a recycle stream of high-boiling oil, was injected through a preheater into the reactor. Cooling hydrogen, in conjunction with external furnaces, controlled the temperature. The products were led into a hot vessel at 425° to 435° C. to remove the highest molecular-weight oils (H.O.L.D.), ash, unreacted coal, and catalyst. Hydrocarbon vapors and gases and excess hydrogen passed to a vessel at room temperature for removing condensable products. The gases were reduced to atmospheric pressure, metered, sampled, and discharged to the atmosphere. The condensable products, after separation of water, were fractionated to produce an overhead oil and a moderately high-boiling fraction, commonly called light-oil bottoms. The latter fraction was blended with H.O.L.D., and the blend, after the solids were removed, was returned to the system for further hydrogenation. Thus overhead oil was the product; its quality and quantity depended on residence time and reaction temperature.

With the small concentration of catalyst, essentially complete conversion of coal resulted, and the hydrogenation of asphaltenes was adequate for satisfactory operation. No difficulties were experienced with temperature control. The asphaltene content of the H.O.L.D. ranged from 33 to 42 percent. Benzene insolubles, including unreacted coal, in the H.O.L.D. remained relatively constant at 9 to 12 percent, regardless of throughput. Yields of total hydrocarbon gas (C_1 to C_6) ranged from 21 to 31 percent, based upon m.a.f. coal; and those of oil boiling under 325° C., ranged from 36 to 51 percent. Coal rates up to 90 p.s.f.h. of reaction space were considerably higher than the 22 pounds employed in German industrial practice with iron catalysts.

Hydrogenation of Coal at 800° C.

Products from the reaction of coal and hydrogen have long been considered as a possible source of gaseous fuels for the future. Appreciable yields of gaseous hydrocarbons can be obtained by hydrogenating coal above 600° C., considerably higher temperatures than those employed for producing liquid hydrocarbons, 450° to 480° C. Studies have been made to determine the effect of short residence time at 800° C. and 6,000 p.s.i. on the production of liquid and gaseous hydrocarbons.

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This work required design and development of apparatus to avoid the long heating and cooling periods inherent in tests made in autoclaves. Long times of heating and cooling superimpose extensive reaction at lower temperatures, especially above 450° C., and consequently do not permit accurate measurement of the effect of residence time.

A simplified flowsheet and details of the reactor are shown in figures 18 and 19. A fixed bed of 30- to 60-mesh coal was supported and retained by

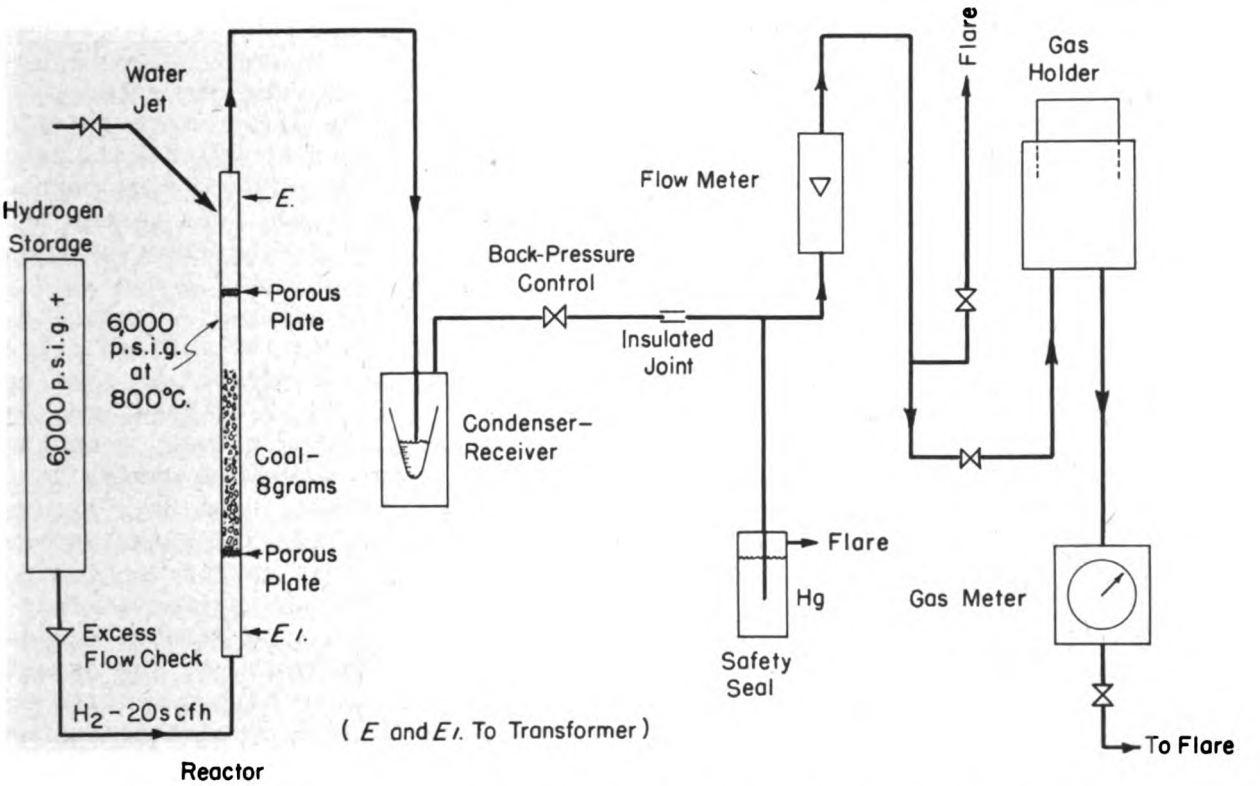

FIGURE 18. - Flowsheet for Hydrogenating Coal at 800° C. and 6,000 p.s.i.

porous plates in a tubular reactor. After establishing a hydrogen flow of 20 std. c.f.h., the charge was heated to 800° C. in about 1-1/2 minutes by passing an electric current directly through the reactor. At the end of the desired reaction time (1 to 15 minutes) the temperature of the reactor and its contents was decreased to room temperature rapidly by quenching with water. High-boiling oils and unreacted solids were washed from the reactor with benzene. The amount of remaining benzene-insoluble solids served to calculate the extent of conversion of coal. Overhead oils were weighed after separation from water and analyzed by a mass spectrometer, as were the hydrocarbon gases. The initial experiments were made with subbituminous coal from Rock Springs, Wyo., impregnated with an aqueous solution of ammonium molybdate to provide a catalyst concentration of 1 percent molybdenum, based upon m.a.f. coal.

At 800° C. and "zero time," the unreacted residue almost equaled the fixed carbon of the coal, but conversion was about 70 percent in 15 minutes at temperature. The bulk of the gases consisted of methane and ethane, and

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the ratio of these constituents varied little with residence time. Total oil yield increased rapidly from 4.6 percent at zero time to 9.0 percent at 1 minute. Although the oil yields remained relatively constant as residence time was increased, the fraction of lower-boiling oil increased. These lower-boiling oils were highly aromatic; the single-ring aromatics were largely benzene and toluene. The concentration of benzene in overhead oil was as much as 58 volume-percent. About 3 to 6 weight-percent of C_6 to C_{10} aromatics and 2 to 3 percent naphthalene were recovered.

Fischer-Tropsch and Related Syntheses

Catalyst Preparation and Pretreatment

Several studies were made of reduced fused-iron catalysts converted to relatively pure interstitial phases. Cementite, prepared by direct carburization and also by thermal reaction of Hägg iron carbide and iron, was tested in the Fischer-Tropsch synthesis at 7.8 and 21.4 atmospheres. Cementite was active and had catalytic properties similar to those of Hägg iron carbide; moreover, cementite was somewhat easier to prepare than Hägg iron carbide. Earlier German investigations indicated that cementite was inactive as a Fischer-Tropsch catalyst. At 7.8 atmospheres both cementite and Hägg iron carbide are usually more active and oxidize less rapidly than reduced catalysts; however, at 21.4 atmospheres, the activity and rate of oxidation of carbided and reduced catalysts were about the same.1/

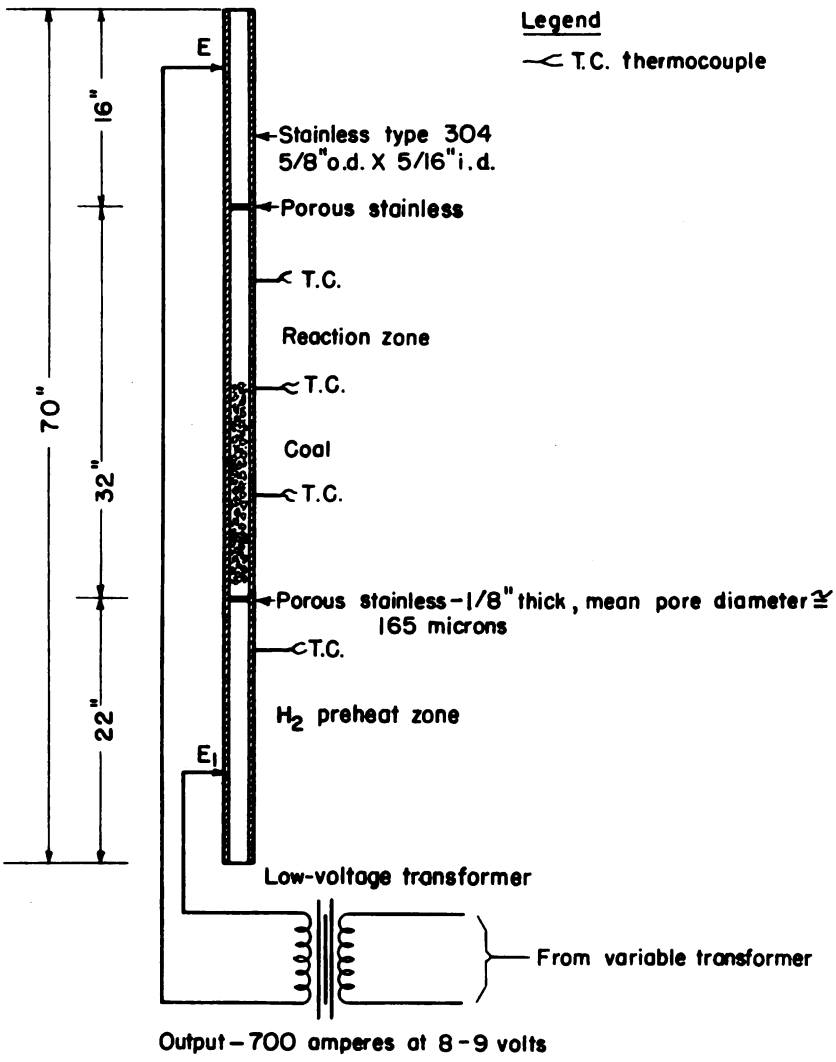

FIGURE 19. - Reactor for Hydrogenating Coal at 800° C. and 6,000 p.s.i.

Two massive iron catalysts were prepared from pure analytical-grade iron wire of .005-inch diameter to provide a catalyst of known geometry containing negligible impurities. As short pieces of wire can be fashioned into

1/ Schultz, J. F., Hall, W. K., Dubs, T. A., and Anderson, R. B., Studies of the Fischer-Tropsch Synthesis. XV. Cementite as Catalysts: Jour. Am. Chem. Soc., vol. 78, 1956, pp. 282-285.

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aggregates of almost any desired shape by the techniques of fiber metallurgy, these tests served to assay the basic activity of wire. The wire was wound into cylindrical form to fit a small reactor tube; catalyst a was wound around a paper core, and catalyst b around a stainless-steel rod. Wire a was packed less closely and weighed only about half of wire b. Subsequent treatment - oxidation with steam at 600° C. - destroyed the paper core but not the steel core. After oxidation the catalysts were impregnated with alkali and reduced in hydrogen. The activity of catalyst a per gram of iron was about twice that of catalyst b, probably due to the hollow core and looser packing. The activities per unit volume were about the same as that of oxidized-steel turnings. After being used in the synthesis, catalyst b was withdrawn and cut into four sections, and each was analyzed for iron, carbon, and oxygen. Carbon was distributed evenly throughout the catalyst bed. The atomic oxygen-iron ratio of the exit portion was about 0.4, compared with 0.03 for the inlet portion. Thus the rate of oxidation during synthesis increased as concentrations of water and carbon dioxide in the gas stream increased. The experiments also demonstrated that chemically promoted iron of high purity is active in the synthesis; structural promoters are apparently unnecessary in massive iron catalysts.

In the hot-gas recycle process (see below) the pressure drop in the catalyst bed must be as low as possible. Studies are in progress to determine how well catalyst cartridges of various shapes meet the dual requirements of high productivity and low pressure drop. Two such shapes studied are (1) a flattened cylinder of iron-wire screen and (2) an assembly consisting of six steel strips welded in the center at each end and resembling a pinion rod.

The wire screen was oxidized to the usual 20-percent oxide content, impregnated with alkali, and then reduced in hydrogen. Excellent performance was obtained in synthesis, characterized by a low initial temperature (235° C.) and by steady improvement in activity throughout the 6 weeks of testing. Preliminary data on the pinion-rod catalyst indicate good performance at 275° C. with a very thin active surface layer (7.5 percent oxidation).

An investigation is in progress to develop catalysts for use at 300° to 400° C. If synthesis can be accomplished without excessive formation of methane or carbon, several engineering advantages are expected: (1) Increased throughput because of increased reaction rate; (2) greater thermal efficiency because steam is produced at higher temperatures and pressures; and (3) decreased production of wax, with concomitant increase in the yield of liquid fuels. At these temperatures the usual carbon-steel reactor shows appreciable activity. This difficulty was eliminated by substituting reactor tubes of type 347 stainless steel or using carbon steel with a copper liner. With these inert reactors special care must be taken to avoid traces of active iron compounds that may be introduced inadvertently, for example, from a previous experiment. Thus, 0.2 gram of iron on a bed of glass wool was moderately active at 355° C., even though the weight of iron was only about 0.2 percent of that used in a typical test at lower temperatures. In a clean reactor materials such as glass wool and refractory silica-alumina support were inactive in this temperature range.

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For producing methane by hydrogenation of carbon monoxide, partly-extracted Raney nickel is the best known catalyst for yield, life, and operating characteristics.2/

Sulfur Poisoning of Iron Catalysts

The sensitivity of a Fischer-Tropsch catalyst to sulfur poisoning determines how completely sulfur must be removed from synthesis gas. Studies of this sensitivity were made by introducing sulfur compounds into synthesis gas and by dissolving them in heptane and impregnating them on the reduced catalyst.

The first method duplicates usual conditions of catalyst poisoning, except that, to accelerate poisoning, more sulfur was put into the gas than normally would be expected. With hydrogen sulfide, the catalyst was inactivated when 1 mg. of sulfur per gram of iron had accumulated on the catalyst; with sulfur dioxide, only 0.1 mg. of sulfur per gram of iron was needed. The poisoning action of sulfur dioxide was unexpected, as sulfur dioxide is only a temporary poison in ammonia synthesis where similar catalysts are used.

The second method was used to show the effect of a sulfur poison when distributed evenly over the catalyst. Six milligrams of sulfur as hydrogen sulfide or four mg. of sulfur as sulfur dioxide per gram of iron was required for essentially total deactivation. This method required more sulfur than the other method to produce a given effect, possibly because the sulfur compounds penetrated deeply into the catalyst pores to sites that are never active in synthesis.

Several attempts were made to reactivate poisoned catalysts by increasing the synthesis temperature to 400° C. Satisfactory yields of hydrocarbons, mostly gaseous, were achieved for several weeks at temperatures between 350° and 400° C. Progressive removal of sulfur was indicated by the odor of mercaptans in the exit gases; however, chemical analyses of the used catalysts indicated that most of the sulfur was retained. Poisoned catalysts were operable above 300° C. for several weeks before becoming plugged by carbon deposits. Thus sulfur compounds appear to poison catalysts for carbon-forming reactions as well as for the Fischer-Tropsch synthesis.

Characterization of Synthesis Products

Gas chromatography has been applied to the characterization of Fischer-Tropsch products, particularly to the C_4 and C_5 hydrocarbon fractions. A detailed investigation of branching in these fractions provides information regarding the growth of the carbon chain during the synthesis reaction.

Branching is fairly independent of pressure, temperature, and conversion. Over a wide range of operating conditions, branching in the C_4 fraction was about 9 percent and in the C_5 fraction about 20 percent. The extent

2/ Schlesinger, M. D., Demeter, J. J., and Greyson, M., Catalyst for Producing Methane From Hydrogen and Carbon Monoxide: Ind. Eng. Chem., vol. 48, No. 1, 1956, 68-70.

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of branching in the C_4 and C_5 fractions follows the chain growth pattern postulated earlier by Bureau workers. Unsaturation also increased as the temperature increased.

With supported iron catalysts, the type of support may materially change the degree of branching. For example, several iron preparations on a commercial cracking catalyst yielded unusually high proportions of branched hydrocarbons in the C_5 fraction, indicating that the support may have had a secondary effect on the composition of the products.

Mass spectra of various groups of oxygenated compounds have been determined for purposes of analysis and for determining the effect of molecular structure on the fragmentation of molecules by electron impact.3 4 5/ Correlating alcohols permits identification of primary, secondary, and tertiary alcohols and gives information on the branching of alkyl chains. Correlating ketones permits identification of the size and configuration of groups adjacent to the carbonyl group. Similar information is obtained by correlating mass spectra of acetals, esters, and trimethylsilyl ethers. In addition to the normal spectral peaks obtained from expected fragmentation by rupturing one bond, so-called rearrangement peaks result by rupturing more than one bond. Hydrogen atoms tend to migrate from the alkyl portion to the oxygenated portion of the molecule. Rearrangement peaks in oxygenated compounds are convenient "fingerprints" for identifying particular groups.

Trimethylsilyl ether derivatives are valuable aids in mass spectral analyses of many compounds containing active hydrogens such as alcohols, glycols, phenols, mercaptans, and amines. The mass spectra of alcohols are very similar to the mass spectra of olefins contained in Fischer-Tropsch products. Converting alcohols to trimethylsilyl ethers permits identification of the original alcohols.

A high-temperature mass spectrometer has been used to obtain spectra of high-boiling compounds. The instrumental requirements are a heated sample-inlet system, a mass-spectrometer tube with small slits, and a stronger magnetic field. The ion source must be cleaned frequently. The equipment was used to analyze alcohols in Fischer-Tropsch products after conversion to their trimethylsilyl ethers. Alcohol yields from three different Fischer-Tropsch processes are compared in figure 20.

A method has been devised for analyzing high-boiling products for alcohols, paraffins, olefins, and ketones. The product is treated with trimethylsilyl ether, phenylhydrazine or carbonyl reagents, and hydrogen. Four mass spectra are required; analyses are made by comparing spectra before and after chemical treatment. Rearrangement peaks of the esters present serve

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3/ Friedel, R. A., Shultz, J. L., and Sharkey, A. G., Jr., Mass Spectra of Alcohols: Anal. Chem., vol. 28, No. 6, 1956, pp. 926-934.

4/ Sharkey, A. G., Jr., Shultz, J. L., Friedel, R. A., Mass Spectra of Ketones: Anal. Chem., vol. 28, No. 6, 1956, pp. 934-940.

5/ Friedel, R. A., and Sharkey, A. G., Jr., Mass Spectra of Acetal-Type Compounds: Anal. Chem., vol. 28, No. 6, pp. 940-944.

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as internal standards to eliminate variations resulting from instrument instability and dilution effects. All of the several steps in the procedure were investigated by analyses of synthetic blends. This method has been applied to a Fischer-Tropsch fraction boiling at 200° to 250° C. Distributions obtained for olefins, paraffins, ketones, and alcohols are shown in figure 21. Esters were detectable but have not yet been analyzed quantitatively.

Routine mass spectrometric gas analyses are now calculated on the Univac electronic computer. Simultaneous equations for 16 component mixtures can be inserted in the computer, all copying of data for calculating and reporting is eliminated, and man-time per sample has been decreased from 35 to 10 minutes.

Chemistry of Carbon Monoxide and Metal Carbonyls

The oxo reaction was reviewed recently.6/ On the basis of kinetic and chemical evidence a mechanism has been proposed for the cobalt-catalyzed addition of carbon monoxide and hydrogen to olefins.7 8 9/ The essential steps are (1) formation of a cobalt carbonyl-olefin complex (2) reaction of this complex with hydrogen, and (3) subsequent formation of an aldehyde by decomposition of the hydrogenated complex.

These reactions provide an insight into the nature and role of surface intermediates in the Fischer-Tropsch reaction, and chain-lengthening may occur by reaction of similarly activated and oriented molecules on the metal surface.

As iron catalysts promoted with alkali usually are used in the Fischer-Tropsch synthesis, an investigation of the chemistry and catalytic properties of the iron pentacarbonyl-aqueous alkali system was initiated.10 11/ Solutions obtained by treating $Fe(CO)_5$ with aqueous alkali serve to convert olefins to the next higher alcohols and to reduce acetylene to ethylene.

6/ Wender, Irving, Here Are Complete OXO Reaction Data: Petrol. Refiner, vol. 35, No. 12, December 1956, pp. 197-200.

7/ Wender, I., Metlin, S., Ergun, S., Sternberg, H. W., and Greenfield, H., Kinetics and Mechanism of the Hydroformylation Reaction. The Effect of Olefin Structure on Rate: Jour. Am. Chem. Soc., vol. 78, No. 20, 1956, pp. 5401-5405.

8/ Greenfield, H., Wender, I., and Wotiz, J., The Reaction of Allenes With Dicobalt Octacarbonyl: Jour. Org. Chem., vol. 21, No. 8, 1956, pp. 875-878.

9/ Greenfield, H., Sternberg, H. W., Friedel, R. A., Wotiz, J. H., Markby, R., and Wender, I., Acetylenic Dicobalt Hexacarbonyls, Organometallic Compounds Derived From Alkynes and Dicobalt Octacarbonyl: Jour. Am. Chem. Soc., vol. 78, No. 1, 1956, pp. 120-124.

10/ Sternberg, H. W., Friedel, R. A., Markby, R., and Wender, I., On The Formation, Structure and Properties of an Iron Carbonyl-Acetylene Complex Prepared by Reppe and Vetter: Jour. Am. Chem. Soc., vol. 78, No. 15, 1956, pp. 3621-3624.

11/ Sternberg, H. W., Markby, R., and Wender, I., Chemistry and Catalytic Properties of the Iron Pentacarbonyl-Aqueous Alkali System: Jour. Am. Chem. Soc., vol. 78, No. 21, 1956, pp. 5704-5705.

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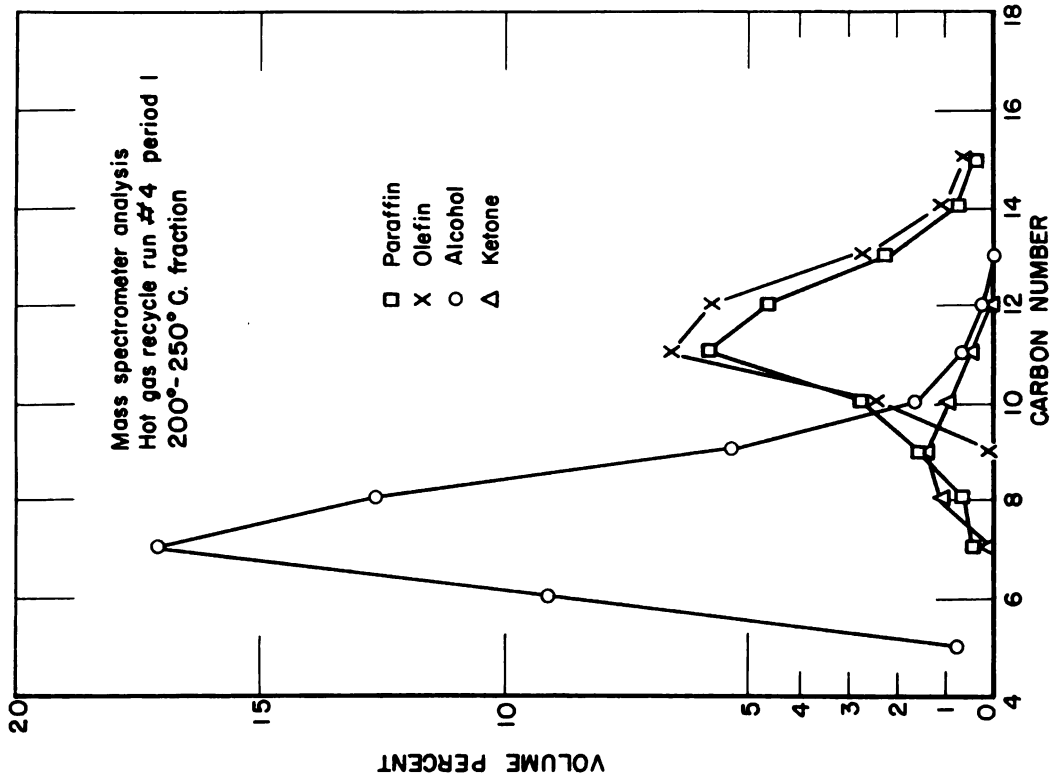

FIGURE 21. - Carbon Number Distribution of Compounds From the Fischer-Tropsch Synthesis.

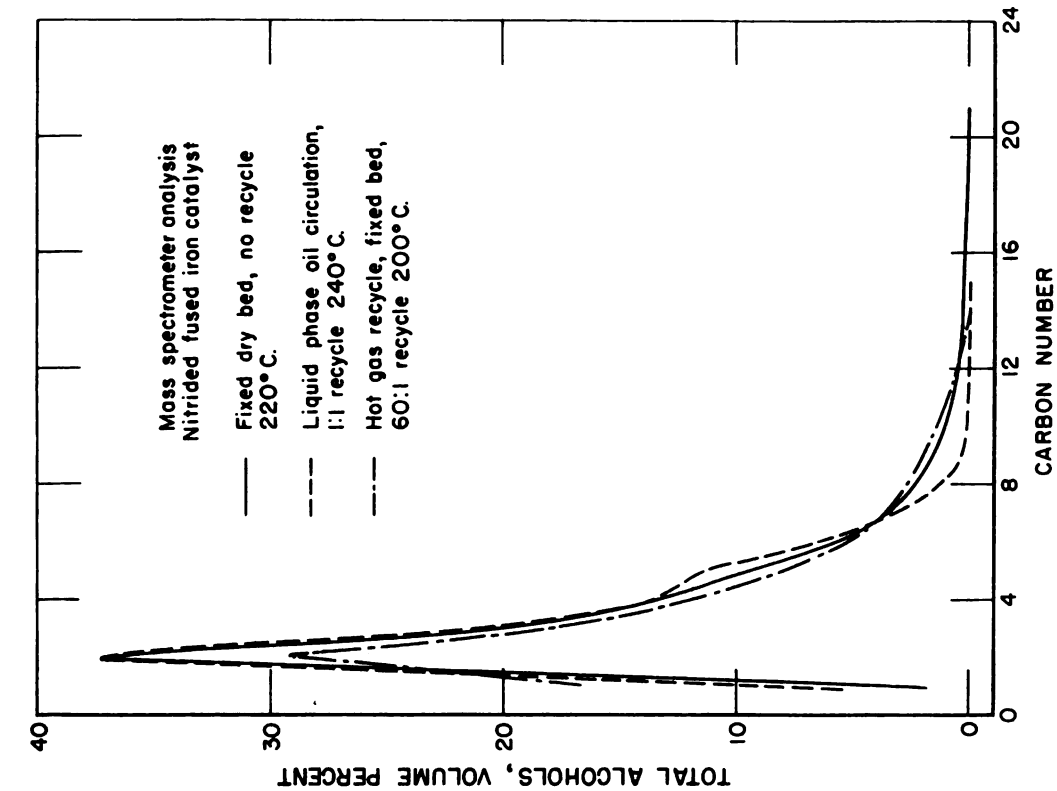

FIGURE 20. - Distribution of Alcohol From the Fischer-Tropsch Synthesis.

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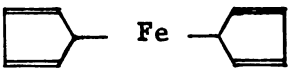
These properties can be explained and new reactions predicted on the basis of the existence of a dimeric ion formed from two $[HFe(CO)_4]$ ions, which decomposes and acts as a hydrogen donor in the presence of a hydrogen acceptor.

When 1-hexene was shaken with a solution containing $[HFe(CO)_4]$ ion for 24 hours at room temperature, 90 percent was isomerized to 2- and 3-hexene. Treating excess cyclopentene with an aqueous solution containing $[HFe(CO)_4]$ ions at 155° C. and 160 atmospheres of CO yielded 33 percent cyclopentanecarboxaldehyde. This is the first report of an iron-catalyzed conversion of an olefin to the next higher aldehyde. The conversion of olefins to the next higher alcohols in the $Fe(CO)_5$-aqueous alkali system now can be explained as taking place in two steps, that is, formation of the next higher aldehyde followed by hydrogenation to the alcohol. That aldehydes are reduced by solutions that catalyze the conversion of olefins to alcohols was demonstrated by treating benzaldehyde with a solution containing $[HFe(CO)_4]$ ion. Benzyl alcohol was obtained in 33 percent yield.

The catalytic properties of a new type of metallo-organic complex formed from transition metals and cyclopentadiene have been investigated. Ferrocene,



is remarkably stable and of little promise as a catalyst. No reaction occurred when ferrocene was treated with CO and H_2 at elevated pressures and temperatures up to 300° C.; nor did ferrocene catalyze the hydroformylation reaction. In fact the compound was recovered essentially unchanged in both instances. In contrast dicyclopentadienyldi-iron tetracarbonyl, which resembles dicobalt octacarbonyl and the similar iron carbonyl complex functions as a catalyst for the hydrogenation of anthracene to dihydroanthracene. In addition, $Fe(CO)_5$ catalyzes the addition of CO and H_2 to olefins at 180° to 200° C.; this suggests that the same mechanism of chain lengthening may be operative with all the usual Fischer-Tropsch catalysts.

The molecular structures of chromium hexacarbonyl $Cr(CO)_6$ and of the ion $Fe(CO)_6$ have been studied by infrared spectrometry.12/ The occurrence of one intense CO-stretching vibration band for both substances indicates that the molecular structures are regular octahedrons.

Preparation of High-Purity Compressed Carbon Monoxide

Commercially available carbon monoxide contains many impurities, including sulfur compounds, which make its use in catalytic processes undesirable. A simple method has been developed for preparing high-purity compressed carbon monoxide. This method consists of condensing carbon monoxide at liquid-air temperatures from readily available sulfur-free $1H_2$+3CO synthesis gas.

12/ Shufler, S. L., Sternberg, H. W., and Friedel, R. A., Infrared Spectrum and Structure of Chromium Hexacarbonyl, $Cr(CO)_6$: Jour. Am. Chem. Soc., vol. 78, No. 12, 1956, pp. 2687-2688.

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After hydrogen and other more volatile constituents have been removed, liquid carbon monoxide is evaporated directly into cylinders.13/

Pilot-Plant Development of Gas-Recycle Process

The heat of reaction of the Fischer-Tropsch synthesis can be removed by recycling large volumes of gases over the catalyst. Circulating gases get 10° to 20° (C.) hotter by contact with the catalyst and are then cooled by generating steam. High operating temperatures that favor maximum yields of gasoline can be tolerated more readily in a gas-recycle process than in an oil-cooled one, and greater space-time yields are obtainable. This method of synthesis was studied first by Michael in Germany at recycle-to-fresh-feed ratios of about 100 to hold the temperature increase through the catalyst bed at 10° C. The catalyst was cube-shaped and offered too high a resistance to the flow of gas. Apparently to minimize the pressure drop, which directly affects compression costs, reactors with a low height-to-diameter ratio were used. As a result nonuniform distribution and flow of gas through the catalyst occurred, and hot spots developed. The project was stopped after a few years because of operating difficulties that seemed insurmountable.

After the Bureau of Mines developed steel lathe-turnings as catalysts in the oil-circulation process, these turnings were investigated in the gas-recycle system. The turnings appeared well suited for this operation, as their large volume of voids (85 to 95 percent) presented low resistance to flow. Because a compressor was not available immediately for recycling hot gases, the operation had to be modified from that normally used in the hot-gas-recycle system. The hot gases leaving the reactor were cooled to room temperature before they entered the compressor and then reheated before being mixed with fresh synthesis gas. As a result, in the studies completed thus far the concentration of water and hydrocarbon vapors in the recycle gas is lower than would be expected if the gas were not cooled. In addition, the reactor had a higher ratio of height-to-diameter than the German one to minimize the danger of a nonuniform flow of gas and consequent development of hotspots.

Lathe turnings of SAE 1018 steel, having a void volume of about 93 percent, were tested in the first experiment. The turnings were oxidized with steam to 20 percent, impregnated with aqueous potassium carbonate, and reduced in hydrogen. Synthesis gas was $1.3H_2$:1CO, and the catalyst bed was 8 feet high; however, to obtain a usage ratio more nearly equal to the feed ratio, a gas richer in hydrogen ($1.5H_2$:1CO) was used during most of the experiment. Carbon dioxide was scrubbed from the recycle gas. During one 24-hour period of steady operation at an hourly space velocity of 1070, 90 percent of the H_2 + CO was converted at an average catalyst temperature of 303° C. About 90 percent of the hydrocarbons above C_2 were in the desired gasoline range; some 9 percent of the total yield was oxygenated compounds, consisting mostly of methyl, ethyl, and propyl alcohols. The yield of C_1 and C_2 gases was not excessive - about 23 percent.

13/ Shultz, J. F., Schoenweis, F. J., and Anderson, R. B., Compressed Carbon Monoxide: Ind. Eng. Chem., vol. 48, No. 8, 1956, p. 1365.

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The recycle ratio was varied from 60 to 40 volumes per volume of fresh feed. Temperature control was excellent; there was no evidence of excessive temperatures. Although the experiment was terminated after only about 1,300 hours of synthesis, total production of hydrocarbons per pound of catalyst was approximately three times that obtained in the oil-circulation process.

Additional experiments were made with a nitrided, granular, fused-iron catalyst. This type of catalyst not only produced hydrocarbons of a lower boiling range than does a reduced catalyst but also larger yields of oxygenated compounds, principally alcohols. Hourly space velocities as high as 750 were investigated, and conversions of $H_2 + CO$ as high as 90 percent were obtained. Synthesis gas ratios, H_2 to CO, of 1.5, 1.3, and 1 were studied, and the maximum operating temperature was 242° C. A pressure of 400 p.s.i.g. and a recycle ratio of 50 were maintained, without removing carbon dioxide from recycle gas. Activity was higher than expected, possibly due to the high linear velocity of the gas. The yield of oxygenates was high, and production of C_1 and C_2 relatively low. At an hourly space velocity of 750, a temperature of 224° C., and a gas conversion of 70 percent, the yield of oxygenates was only slightly below that obtained at an hourly space velocity of 200 at 200° C. and the same conversion; however, at 90 percent conversion of $H_2 + CO$ and 242° C., the yield of oxygenates was lowered considerably. No operating difficulties were encountered.

Below is a comparison of products obtained with a nitrided catalyst in the fluid-bed, slurry, and cold-gas-recycle processes. The fluidized bed and cold gas recycle produced about the same amounts of oxygenated compounds, but the latter yielded only about 60 percent as much C_1 and C_2 gases.

	Fluidized bed	Slurry	Cold-gas recycle
Temperature.....° C.	251	237	228
Conversion..... percent	68.0	69.2	69.5
Yields:			
$C_1 + C_2$ gases.....weight-percent	31.5	28.9	18.1
C_{3+} hydrocarbons.....do.....	28.2	38.3	40.0
Oxygenated compounds (excluding H_2O and CO_2).....	40.3	32.8	41.9

On the basis of results obtained thus far, removing heat by means of recycle gases appears promising and merits further research. The high linear velocity of gas through the catalyst bed, up to 8 feet per second, apparently increases the activity of the catalyst. Possibly thinner liquid films cover the surface of the catalyst, so that diffusion of the gases both to and from the surface of the catalyst is more rapid.

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Review Articles Relating to Fischer-Tropsch Synthesis

A series of review articles have been contributed to a treatise on catalysis.[14] These articles constitute almost the whole of volume IV, and together they represent a comprehensive evaluation of our knowledge of Fischer-Tropsch catalysis as of 1956. The various chapters are as follows:

Chapter I. - The Thermodynamics of the Hydrogenation of Carbon Monoxide and Related Reactions, by R. B. Anderson. Summarizes the thermodynamics of many organic reactions that occur in the Fischer-Tropsch synthesis, as well as some related ones. Presents the best available thermodynamic data for the solid phases, such as carbides, oxides, and nitrides. Discusses the thermodynamics of iron and nickel carbonyls.

Chapter II. - Catalysts for the Fischer-Tropsch Synthesis, by R. B. Anderson. Contains a history of the development of Fischer-Tropsch catalysts and the processes in which they are used. Presents details of preparing, testing, and evaluating catalysts. Discusses factors influencing activity, selectivity, and stability.

Chapter III. - Kinetics and Reaction Mechanism of the Fischer-Tropsch Synthesis, by R. B. Anderson. Presents data pertinent to the kinetics and mechanism of the hydrogenation of carbon monoxide, the hydrogenation of carbon dioxide, and the water-carbon monoxide reactions. A theory of the growth of hydrocarbon chains in the Fischer-Tropsch synthesis is developed, but kinetics, selectivity, promoter effects, etc., cannot be predicted yet on theoretical bases.

Chapter IV. - Crystalline Phases and Their Relation to Fischer-Tropsch Catalysts, by L. J. E. Hofer. Examines the catalytic activity of phases of elements active in the Fischer-Tropsch synthesis. Shows a relationship between activity and completion or incompletion of the "d" shell. This relationship may make the search for new catalysts less haphazard.

Chapter V. - The Isosynthesis, by Ernst M. Cohn. Describes the process developed during World War II in Germany for preparing highly branched products from synthesis gas. Describes catalysts, testing equipment, effects or process variables, and product distribution, and considers the mechanism of the reaction.

Chapter VI. - Methanation, by Murray Greyson. Considers the thermodynamics, catalysts, industrial techniques, and kinetics of producing methane by the hydrogenation of carbon monoxide.

14/ Emmett, P. H., Catalysis. Vol. IV: (ed.), Reinhold Pub. Corp., New York, N. Y., 1956, pp. 1-511.

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SCIENTIFIC WRITING

Three publications deal with various phases of writing scientific publications: One paper concerns the purpose and motives in scientific writing; its general characteristics; and the time, place, and consequences of publication.15/ Another bears on the mechanics of assembling a scientific manuscript; it covers gathering and correlating data as well as the actual writing.16/ The third discusses the preparation of graphs; it emphasizes the need for simplicity, clarity, and completeness; it describes briefly the use of less conventional representation by such means as derivatives, logarithmic time scales, and inverse logarithmic scales.17/

15/ Cohn, E. M., Approaches to Scientific Writing: Tech. Writers and Ed. Jour., Fall 1956, pp. 6-8.

16/ Cohn, E. M., Systematic Approach to Manuscript Preparation: Jour. Chem. Ed., vol. 33, No. 10, October 1956, pp. 523-525.

17/ Cohn, E. M., Editing Graphs for Publication: Am. Doc., vol. 6, No. 2, April 1955, pp. 77-86.

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